Metallurgy Department Progress Report for the Period 1 January to 31 December 1986

Risø National Laboratory, Roskilde

Publication date:
1987

Document Version
Publisher's PDF, also known as Version of record

Risø-R-547

RISØ

Risø-R-547

Metallurgy Department Progress Report for the Period 1 January to 31 December 1986

Risø National Laboratory, DK-4000 Roskilde, Denmark
April 1987

Risø-R-547

METALLURGY DEPARTMENT PROGRESS REPORT FOR THE PERIOD 1 JANUARY TO 31 DECEMBER 1986

Edited by A. Schrøder Pedersen and J.B. Bilde-Sørensen

Abstract. The activities of the Metallurgy Department at Risø during 1986 are described. The work is presented in four chapters: General Materials Research, Technology and Materials Development, Chemical and Electrochemical Energy Research and Development, and Fuel Elements. A survey is given of the Department's participation in international collaboration and of its activities within education and training. A list (with abstracts) of publications and lectures by the staff during 1986 is included.

April 1987
Risø National Laboratory, DK-4000 Roskilde, Denmark

ISBN 87-550-1311-2
ISSN 0106-2840

CONTENTS

1. INTRODUCTION

Over the last decade the activities of the Metallurgy Department have gradually changed from being concentrated mainly on nuclear metallurgy to covering a number of different areas within materials science and engineering. This change has been motivated by the diminishing interest in building nuclear power stations in Denmark. At present the Danish energy plans do not include nuclear energy and as a consequence the main objective of Risø's programmes has been changed from nuclear energy research to energy research in general (Law no. 194, 28 April 1986).

The work of the department is presented in this report in four sections: General Materials Research (Chapter 2), Technology and Materials Development (Chapter 3), Chemical and Electrochemical Energy Research and Development (Chapter 4) and Fuel Elements (Chapter 5). The projects extend from fundamental materials research to industrial projects on materials application. Most of the projects relate to energy applications as in fuel element research, fusion technology work, development of light and strong materials and characterization of materials to be used in energy storage systems. Projects of a more general nature are carried out within the areas of fatigue and creep, non-destructive testing, neutron radiography and ceramics. The joint work with the Physics Department on neutron diffraction for texture measurements has been extended to cover also determination of internal stresses in materials and engineering components. An important part of this work has been in-situ measurement of internal stresses in metal-fibre composites.

The many research areas have required procurement of new equipment. Furthermore the automation of existing equipment has been continued. Part of the purchases to the department have been financed through income from contract research, which is now covering about 40 per cent of the total activities. From a pri-

vate foundation "Villum Kann Rasmussen Fonden", the department received as a gift a 200 kV transmission electron microscope, which was delivered at the end of the year.

The department participated in international collaboration on specific research projects and in study groups under the auspices of EEC, NEA, IAEA and various Nordic and US organizations (Chapter 6). The department organized the 7th Risø International Symposium on Metallurgy and Materials Science, 8-12 September 1986. The title of this symposium was "Annealing Processes - Recovery, Recrystallization and Grain Growth". The symposium was attended by approximately 100 participants and 73 papers were presented. Planning was started for the 8th Risø International Symposium to be held at Risø 7-11 September 1987 on "Constitutive Relations and Their Physical Basis". Educational activities were continued, students and graduates from Denmark and abroad studied in the department.

2. GENERAL MATERIALS RESEARCH

The materials research programme includes long-term experimental and theoretical studies aimed at contributing to an understanding of the physical mechanisms governing the properties of materials of general technological interest. A large part of this work is carried out in collaboration with universities and research laboratories in Denmark and abroad. Major efforts are devoted to materials preparation and testing and to microstructural characterization by a variety of microscopical and non-microscopical techniques. The relation between the observed macroscopic and microscopic behaviour is studied theoretically in terms of crystal lattice defects by computer methods and combinations of continuum models and discrete models. The materials under investigation comprise metals, alloys and composite materials. The research results are published in the open literature.

2.1. Recrystallization Kinetic Models

Grains of different orientations may nucleate and grow differently. This has been incorporated in a computer model for recrystallization. The model has been used to simulate the recrystallization behaviour of heavily deformed aluminium. In this material grains of cube, rolling and random orientations develop differently. In the model the grains are assumed to nucleate instantaneously or after an initial incubation period and to grow spherically with a constant growth rate until impingement. When two grains impinge, they stop growing in that direction, but continue to grow freely in all other directions. As input for the model experimentally measured values for the initial incubation periods and the growth rates for the three types of grains were used. The calculated results for the volume fraction of recrystallized material, the average grain size of the three types of grains as a function of annealing time and the grain size distribution in the fully recrystallized state were compared with ex-

perimental results. In general reasonable agreement between model calculations and experimental measurements were obtained. The main discrepancies were that the model overestimates the average size of the small random grains and underestimates the average size of the large cube grains at the end of recrystallization (Figure 1). This can be understood if, in a real material, some

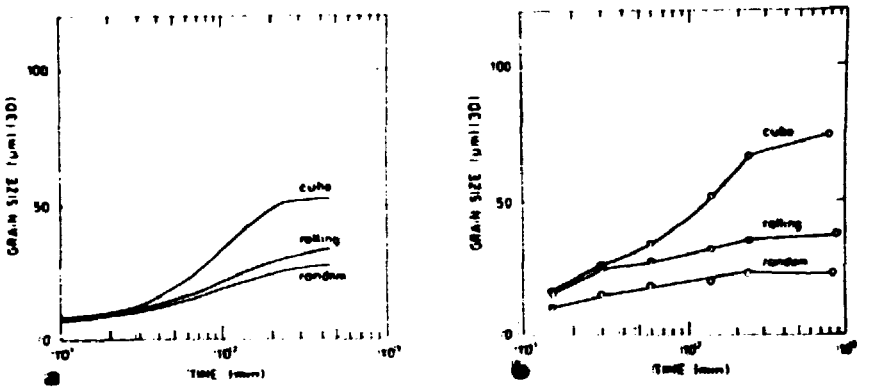

Fig. 1: The average grain diameter (in 3D space) of the three texture components. (a): Calculated results. (b): Experimental results determined by SEM.

grains can cease growing without being stopped by impingements, and some grains can grow at the expense of already existing grains as for instance observed by in-situ high voltage electron microscopy investigations.

Another computer program was developed for a general investigation of nucleation and growth. With this program it is possible to simulate the effect of different nucleation and growth mechanisms on the recrystallization process. An example is shown in Figure 2. Here it is assumed that nucleation takes place with a constant nucleation rate in the unrecrystallized material and that the grains grow with a constant volume growth rate. The volume fraction of recrystallized material is calculated as a function of time (shown by circles in Figure 2) and the result

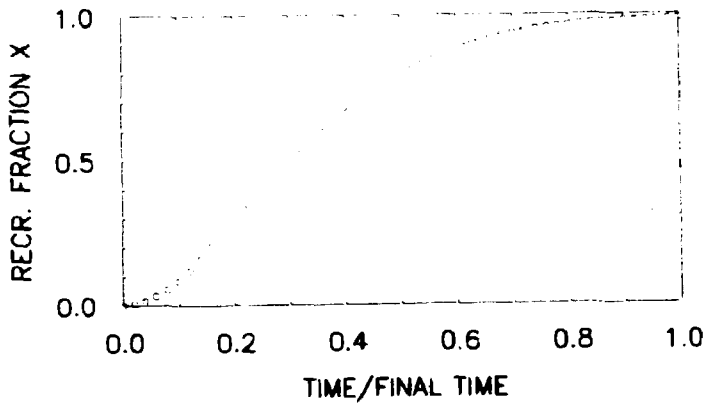

Fig. 2. The volume fraction recrystallized versus time for a recrystallization process with a constant nucleation rate and a constant volume growth rate. The full curve is the best Avrami fit to the calculated points.

is fitted to the Avrami equation (shown by the full line in Figure 2). In the Avrami equation the time exponent β describes the shape of the curve. Experimentally β-values are generally found in the range 0.7-2. From the simulation it was clear that β-values in this range cannot be obtained with any of the simulated nucleation modes if the radial growth is linear. In order to simulate recrystallization kinetics with β ~ 1 one has to assume almost instantaneous growth to final size.

2.2. Grain Growth in Pure Copper

The textural and microscopical changes were followed during grain growth in 99.999 wt % pure copper. It was found that the <111> texture strengthens during annealing in the temperature range 425-600°C (Figure 3), whereas the <100> component remains relatively stable. The microscopical investigation showed that during grain growth the grain size distribution broadens and that the larger grains tend to have orientations close to <111>. In a

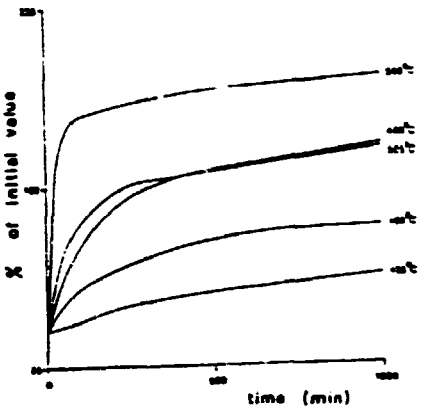

Fig. 3. Kinetic curve showing the variation of the <111> texture component as a function of annealing time.

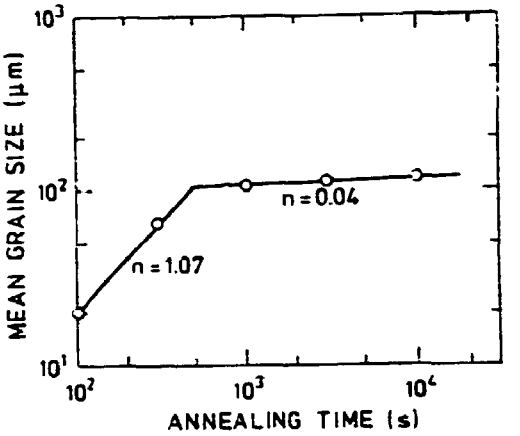

Fig. 4. The increase in mean grain size (log D) as a function of annealing time (log t) for grain growth at 600°C.

plot of the mean grain size D versus annealing time, a large variation in the time exponent n ($D \sim t^n$) was seen. During the initial stage of grain growth the exponent is high (n=1.07) falling to a very low value (n=0.04) for the later stage. Recently computer models have been extended to include the effect of texture, and when the experimental results were compared with the results of these models reasonable agreement was found.

2.3. Effect of Particles on the Deformation and Recrystallization Texture

The investigations of the effect of particles on texture and microstructure in deformed and recrystallized materials were continued. The effect of small particles was studied in a series of aluminium-aluminium oxide specimens containing from $0.16 \cdot 10^{-2}$ to 0.4 vol. percent aluminium oxide particles of a diameter less than 250 nm. The particle spacing varied within an order of magnitude, and the textural changes after cold-drawing (50 and 90 percent) and after recrystallization were analyzed as a function of the spacing.

It was found that a strong <111> fibre texture develops during large deformations and that the strength of the texture varies with the spacing. This effect is related to the change in slip pattern caused by the particles.

The recrystallized texture is weaker than the deformation texture and is a mixed <100> - <111> texture. In the recrystallized state the concentration of the <111> component depends on the particle spacing in such a way that its decrease during recrystallization gets smaller when the spacing is decreased, i.e. the cold-drawing texture is retained to an increasing degree with decreasing particle spacing (Figure 5). This observation may be related to an effect of particles on the formation of recrystallization nuclei which may take place at the original high angle boundaries or at segments of high angle boundaries formed in the transition bands.

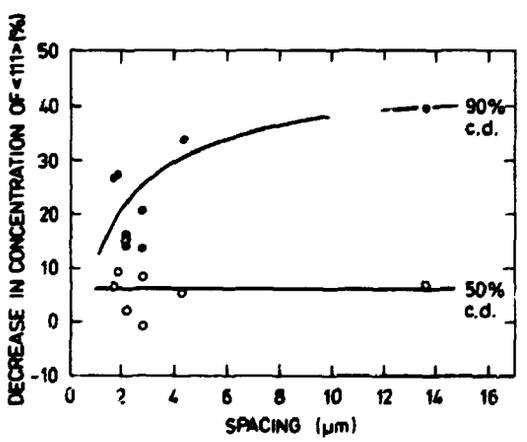

Fig. 5. Decrease in the concentration of the <111> component during recrystallization after cold-drawing as a function of the particle spacing.

2.4. Plastic Deformation of Polycrystals

The textural development and the flow stress was determined for pure copper (99.999%) in compression. For strains below 1.4 the textural development was in qualitative agreement with a Taylor-model prediction, i.e. a maximum concentration at the <110>-component. However, the rate of textural changes and the concentration of the <110>-component were overestimated in the model. For large strains (e=1.4-2.9) the textural development was in broad accordance with relaxed-constraint model prediction.

Flow softening is observed at e > 1.1. This phenomenon is related to the formation of a relatively strong <110> texture. In this case the slip systems are oriented in such a way that further slip is by plane strain. This type of deformation requires less plastic work than the normal axisymmetric deformation, and may therefore cause softening. In the plane strain mode compatibility between individual grains can only be obtained if the grains bend around each other, the so-called curling. A metallographic examination of specimens compressed to a strain of 2.9 shows that curling takes place in these specimens.

The study of the effect of initial grain size on the textural and microstructural development was continued. In two series of cold rolled aluminium specimens it was found that the rate of textural development during deformation decreased with increasing initial grain size and even at high strain (cold rolled 90%) the intensity of the main rolling components was lower in the coarse grained materials. These findings were typical both for specimens with a strong and with a weak starting texture. Microscopically it was observed that the deformation pattern (specially at low and medium degrees of deformation) is more homogeneous in fine-grained than in coarse grained specimens. It was suggested that this more homogeneous deformation pattern is connected to the faster texture development in the fine-grained specimens.

The effect of a starting texture on the textural development was investigated by a computer procedure where the measured starting

textures were transformed to a collection of grain orientations (~ 10000) and with a texture simulation program the development of these grain orientations were followed. The program was used to include the starting texture in a simulation of the textural development in the fine- and coarse-grained aluminium specimens described above. An example of the agreement between measurement and simulation is shown in Figure 6. Here the measured and the

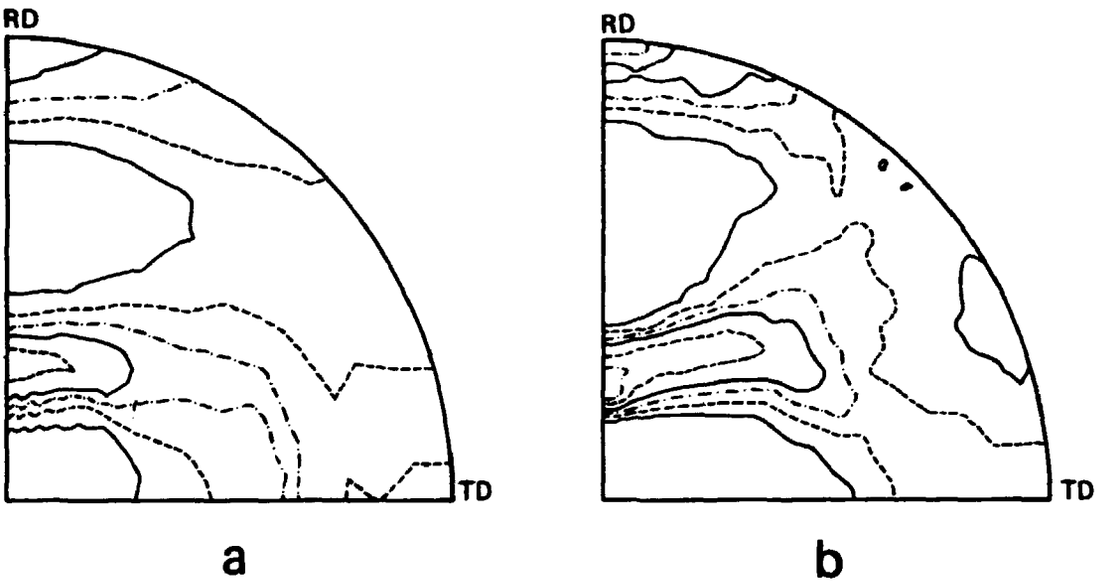

Fig. 6. (111) pole figures for fine grained aluminium cold-rolled to 50% reduction in thickness. (a): Measured pole figure. (b): Simulated pole figure.

simulated (111) pole figure at 50% reduction in thickness is shown for fine-grained aluminium with a weak starting texture. By comparing measured and simulated textures it was concluded that the observed differences in texture development between fine- and coarse-grained aluminium specimens were not just trivial effects of differences in starting texture, but genuine grain size effects.

2.5. Constitutive Equations for Steel

Constitutive laws for steel loaded into the plastic range by cyclic thermal and mechanical loads were studied theoretically and experimentally in collaboration with a major industrial company. The aim of the project is to develop and verify one or two constitutive laws for two specific materials, a CrMo-steel used in engine components and a stainless steel, and to implement the laws in existing finite element codes. The model shall cover the material in the temperature range 20 to 400°C, and as the laws shall model the operational conditions for the components, the strain range can be limited to a few percent. Work on the project was initiated in late 1986 with a literature survey, adaption of an existing model to a new computer installation, preliminary tests on the chosen material and planning and purchasing of equipment for the experimental part. Both uniaxial and biaxial tests will be necessary, and the final verification will involve measurement of residual stresses by neutron diffraction in a model component.

2.6. Fatigue in Metals

Studies of the mechanisms of flow, structural change and fracture in simple histories of low-temperature monotonic and cyclic deformation were continued. Each mechanism is restricted to a particular field in a "fatigue diagram", a plot of the plastic strain amplitude versus the cycle number. The construction of fatigue diagrams was completed for single crystals and polycrystals of copper. Tentative diagrams were constructed from recent data on Al and Mo to examine the effects of lattice structure, temperature and strain-rate on the diagram.

The fatigue diagrams can provide guidance in interpreting fatigue processes in materials and deformation histories more complicated than single-phase metals deformed in tension-compression at constant plastic strain amplitudes. In an application of the diagrams available data for copper deformed at constant stress amplitudes in tension-compression or at constant total strain am-

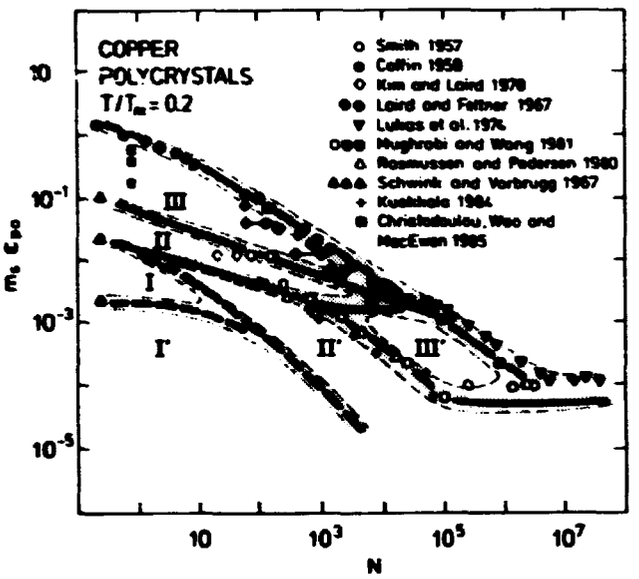

Fig. 7a. The fatigue diagram for copper polycrystals displays a pattern of fields, which represents the successive stages of cyclic hardening and fatigue at constant plastic strain amplitudes. The plastic shear amplitude, $m_s \varepsilon_{pa}$, is plotted versus the cycle number N.

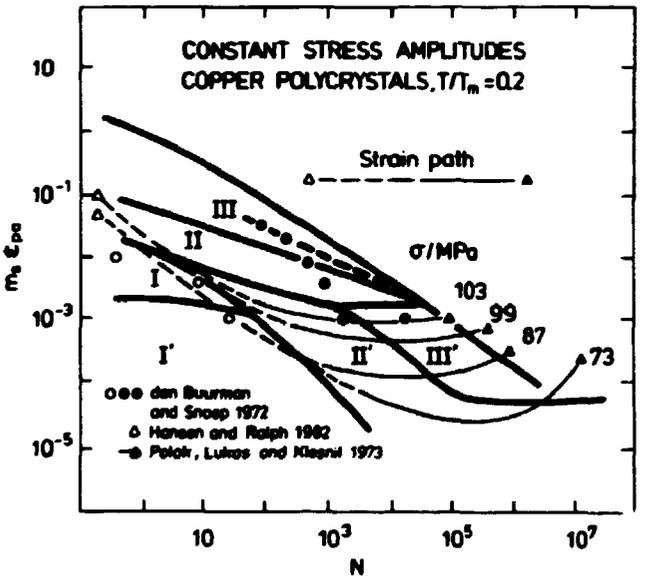

Fig. 7b. The fatigue diagram can be applied in interpreting fatigue under variable plastic strain amplitudes in tension-compression or torsion.

plitudes in torsion were plotted onto the fatigue diagram for copper. Observed changes of plastic strain amplitude, elastic modulus and internal friction were found to be correlated with the pattern of fields in the fatigue diagram.

Experimental studies of the work hardening and fatigue in pure copper and copper-tungsten composites were continued. The effects of elastic heterogeneity, strain hardening and plastic relaxation on the constitutive behaviour were measured in cyclic experiments

at 293 K and 77 K. A study was initiated to examine the distribution of persistent slip bands in single slip oriented copper-tungsten.

2.7. Irradiation Experiments With 600 and 800 MeV Protons

(In collaboration with EIR/SIN, Würenlingen, Switzerland, Los Alamos National Laboratory, New Mexico, U.S.A. and IFF-SNQ, KFA Jülich, FRG).

Specimens (3mm discs, 0.15 mm thick) of pure copper, aluminium, Al-0.75% Mg-0.42% Si alloy and Al-1.2% Mg alloy were irradiated with 800 MeV protons in the Los Alamos Meson Physics Facility (LAMPF) at Los Alamos National Laboratory. A total of 26 discs were irradiated to doses between 0.1 and 4 displacements per atom (dpa). The irradiation was carried out at 40°C. Together with these discs, thin (0.25-0.3 mm) tensile specimens of commercial AlMg3 and Al-6061 (Al-Mg-Si) alloys were also irradiated at 40°C. The specimens of AlMg3 alloy were irradiated in the annealed as well as 50% cold-worked conditions whereas the specimens of Al-6061 alloy were irradiated in the solution annealed and aged (to peak hardness, T6) conditions.

The microstructure of the irradiated disc specimens is being investigated by transmission electron microscopy (TEM) and positron annihilation technique (PAT).

Tensile properties of the irradiated as well as unirradiated AlMg3 and Al-6061 alloys were determined at 20, 100, 200 and 300°C. Electrical resistivity of the irradiated and unirradiated AlMg3 and Al-6061 specimens was determined at 20, 50, 100, 150 and 200°C. Results of these measurements are being evaluated.

An irradiation experiment with spallation neutrons in Spallation Neutron Area at LAMPF was initiated; the experiment will continue during 1987. In this experiment a number of Cu and Cu-alloys specimens (3 and 8 mm discs) are being irradiated at 330 and 400°C. In the same experiment, 3 mm disc specimens of pure aluminium, pure Al-Mg alloy, pure Al-Mg-Si alloy and commercial AlMg3 and Al-6061 alloys are being irradiated at 120oC.

Samples of pure Al-0.75% Mg-0.42% Si alloy were solution annealed and irradiated with 600 MeV protons in the PIREX facility at the Swiss Institute for Nuclear Research (SIN). The irradiation experiments were carried out at 169 and 276°C to a dose level of up to 1.87 dpa. The microstructures of these specimens are being investigated by TEM.

2.8. Evolution of Microstructural Inhomogeneity and Formation of Void Hyperlattices During Irradiation

(In collaboration with A.E.R.E. Harwell, England)

The study of the effect of planar sinks (e.g. grain boundaries) on the evolution of microstructural inhomogeneity was continued. High-purity (99.999%) aluminium specimens irradiated with fast neutrons in DR 3 (at Risø) and HFR (at Petten) were investigated by TEM. Both the nucleation and growth of cavities were appreciably enhanced in a relatively wide (of the order of 20 cavity-spacings) band near grain boundaries. The vacancy accumulation was found to peak at a distance of about 10 cavity spacings from the boundary. The cavity density in the peak zone was distributed fairly symmetrically with respect to the peak position. More significantly, the enhanced accumulation of vacancies in the peak zone was found to continue up to high doses (e.g. 2×10^{26} n/m^2 ≈ 19 dpa).

In an attempt to understand the phenomenon, the enhanced accumulation of vacancies in the peak zone and its variation with distance from grain boundaries were calculated analytically for different modes of transport of self-interstitial atoms. It was shown that the conventional three-dimensional diffusion and biased trapping of interstitials cannot predict the observed enhancement and its spatial variations in the peak zone. The long-range chanelling of the displaced atoms into a grain boundary, on the other hand, can produce an enhancement which is in a reasonable agreement with experimental results. The mechanism does require, however, that an appreciable number of atoms (~ 1%) are channelled over distances of several microns.

Void hyperlattices were observed in high-purity aluminium irradiated with fast neutrons at 50°C to a dose level of 2×10^{26} n/m^2. Void ordering was found to occur at a much lower void density and with a void lattice-spacing considerably larger than hitherto reported in the literature. In spite of void ordering, voids appeared to maintain a healthy growth rate. The next-nearest neighbour separation between voids on hyperlattice sites was found to approach 1000 atomic distances. Hence, it was considered to be unlikely that any interaction with this magnitude of separation can be achieved by any other means than transport of point defects within the host lattice. In view of the fact that the void lattice spacing generally decreases with increasing mass density, it was suggested that the formation and stability of void hyperlattices may arise from the dynamic transport (via dynamic crowdions, replacement collision sequences or channelling) properties of self-interstitial atoms.

2.9. Cavity Nucleation and Gas Transport to Grain Boundaries During Irradiation

(In collaboration with A.E.R.E. Harwell, England)

In a previous calculation it was shown that the cavity density observed in high-purity aluminium irradiated with 600 MeV protons could not be explained in terms of helium diffusion via the self-interstitial displacement mechanism or the di-vacancy mechanism. New calculations have been made of the diatomic nucleation of cavities, assuming that helium atoms trapped in vacancies diffuse by a substitutional diffusion mechanism. Within the framework of radiation enhanced diffusion, the effective diffusion coefficient for a helium atom, D_{eff} (He), undergoing substitutional diffusion is found to be proportional to $C_v D_v$ where C_v and D_v are the vacancy concentration and diffusivity, respectively. The magnitude of the calculated cavity densities were found to be very similar to the observed densities in metals and alloys irradiated under the condition of continuous gas production. The temperature dependence of the cavity density was found to be in excellent agreement with that observed in 600 MeV proton irradiated high-purity aluminium.

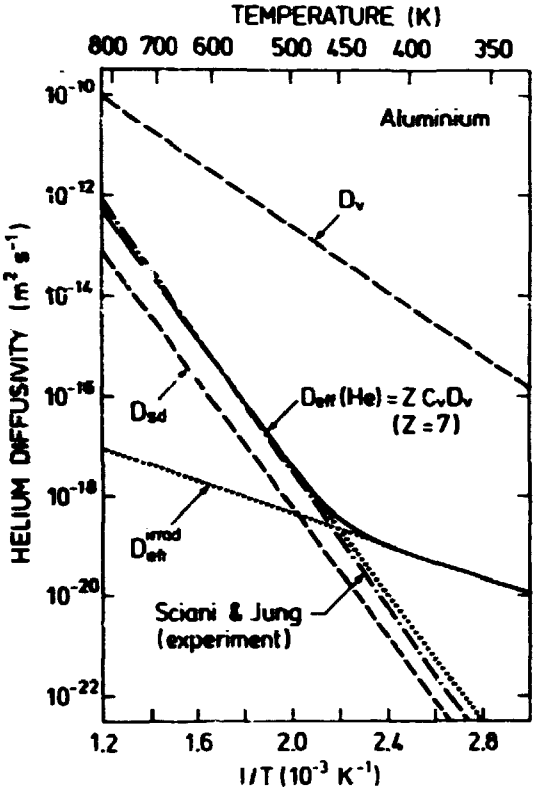

Fig. 8. Temperature dependence of effective helium diffusivity D_{eff} (He) in aluminium, assuming substitutional helium diffusion, shown for a damage rate of 3.5 x 10^{-6} dpa s^{-1} (600 MeV protons). Comparison is made with measured vacancy diffusivity D_v, self-diffusion coefficient D_{sd}, and an experimental determination of helium diffusivity for T > 573 K.

The study of gas transport to grain boundaries during irradiation was continued. The flux of gas atoms reaching a boundary during the nucleation of cavities within the grains was calculated on the basis of a diatomic nucleation model. It was found that because of fast nucleation no great loss of gas to the boundaries should occur during the nucleation period. The gas loss to the boundaries could be considerably enhanced, however, if any delay in bubble nucleation were to occur due to incubation effects. It was pointed out that the gas flux to the boundaries is dependent primarily on the nucleation behaviour of cavities or/and any other sink for the gas atoms in the grain interior. It was concluded that in cases where the diffusion of gas atoms in the grain and the nucleation behaviour of cavities are reasonably well understood, the flux of gas atoms to the boundaries could be predicted with an accuracy of ~ 50%.

2.10. Positron Annihilation Investigations of Defects in Metals

The work has mainly been concentrated on experimental and theoretical studies of voids and rare gas bubbles in metals. This has been done partly in order to establish the positron annihilation technique (PAT) as a quantitative spectroscopy for defects in metals, and partly in order to obtain information about these defects and their annealing behaviour (cavity size and density, density of gas in bubbles). Continuing earlier work, helium bubbles in aluminium and krypton bubbles in copper and nickel were investigated. The He bubbles were generated by 600 MeV (Swiss Institute for Nuclear Research) or 800 MeV (LAMPF, Los Alamos) proton irradiation at temperatures in the range 50-450°C and to doses in the range 0.2-1.3 displacements per atom. The krypton bubbles were created by a combined implantation and sputtering method (Harwell, UK).

Calculations of the relation between positron lifetimes and Kr densities in bubbles in Cu have been carried out (in collaboration with the University of Jyväskylä, Finland). Using this theoretical relation (or the equivalent one derived earlier for He bubbles in Al) together with a semiempirical formula which was established for the positron trapping rate into cavities as a function of cavity size, it was demonstrated that from positron lifetime measurements one can derive bubble sizes and densities as well as gas densities in the bubbles. This is illustrated in Figure 9. Also the shapes of angular correlation curves have been shown to give information about He densities in bubbles. After annealing close to the melting point the Cu and Ni samples contained high concentrations of large Kr-bubbles with low Kr density. At low temperatures the krypton condensed at the bubble surfaces. PAT is particularly sensitive to this condensation and measurements have been initiated (in collaboration with the Technical University of Denmark) to study the condensation in some detail.

As a separate project (in collaboration with the Computer Section, Risø, and supported by the International Atomic Energy Agency) work is going on to modify the widely used main frame

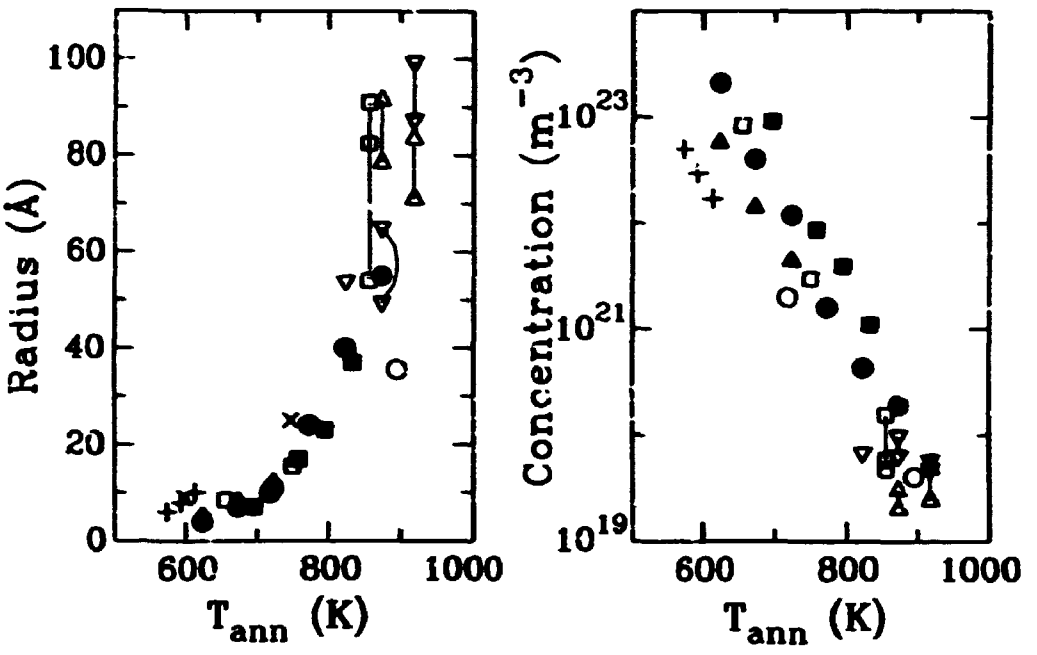

Fig. 9. Radius and concentration of helium bubbles in aluminium as functions of annealing temperature. The closed symbols are results obtained from position lifetime measurements in three different 600 MeV proton irradiated samples. The other symbols are electron microscopy data from the literature for He-implanted Al. All samples were prepared below 200°C before annealing.

computer programs for PAT data analysis previously developed at Risø, in order to make them compatible with modern Personal Computers.

2.11. Creep of Metal Matrix Composites

Improvements in the high temperature mechanical properties of metals and alloys can be achieved by fibre reinforcement. Projects are in progress to study the creep behaviour of aluminium and its alloys after reinforcement with fibres, typically SiC-fibres and Al_2O_3-fibres. A commercial alloy Al2124 (major alloying elements Cu, Mg and Mn which cause precipitation hardening) with 15 and 25 volume percent SiC-fibres was creep tested at 300oC. The creep strengths of the composites were higher than

those of the pure alloy; the increase was, however, less than expected from simple mechanical models for creep of fibrous composites. Furthermore, the stress sensitivity of the creep rate of the composites was very high, and higher than predicted. The reason for these discrepancies was supposed to be a possibly complex interaction between the fibres and the precipitation process in the matrix alloy.

The models for the creep rate of fibrous composite materials describe the dependence on the applied stress (see Progress Report for 1985). The creep rate refers to the steady state rate during the middle period of the creep life time of the material. An extension has been made to describe analytically the creep rate in the final stage of creep leading to failure. The model predicted creep rates which were 3 to 5 orders of magnitude larger than the steady-state creep rates. This final stage of creep is very short for the composites.

2.12. Internal Stresses in Fibrous Composite Materials Measured by Neutron Diffraction

Fibre reinforced materials contain internal stresses caused by thermal and mechanical treatments. The thermal effects are caused by the difference in thermal expansion of the fibres and the matrix, and the mechanical effects are caused by the difference in elastic and plastic properties of fibres and matrix. A study of the internal stresses after various thermal treatments was made; the material was (pure) aluminium with 5 volume percent of SiC-fibres. The thermal treatment was both quenching from temperatures of 400, 300, 200, and 150°C, and continuous heating and cooling between room temperature and 500°C. The internal strains were measured by neutron diffraction techniques; these allow sampling of volumes of the order of cm^3, i.e. a whole specimen.

After quenching the internal strains were tensile in the Al-matrix and compressive in the SiC-fibres of magnitude $+6 \times 10^{-4}$ and -20×10^{-4}, respectively. The strains after quenching corre-

lated well with a (simple) model, based on Eshelby's ideas for matrices with inclusions.

During the heating and cooling treatment, the different thermal expansions of fibres and matrix caused internal strains to be set up. The corresponding (often large) stresses could initiate relaxation processes especially at temperatures above 200°C. The resulting internal stress field was complex, and the numerical values of strains present were typically up to 20 x 10^{-4} in the matrix, and 30 x 10^{-4} in the fibres. A conclusion from the thermal experiments is that at room temperature there is (still) some relaxation (due to high stress levels), and at 500°C there is (still) retained internal strains (due to difficult relaxation processes).

3. TECHNOLOGY AND MATERIALS DEVELOPMENT

The materials technology programme concentrates on the development of new materials and methods for their fabrication, characterization, testing and design applications. Most of the projects are carried out in collaboration with the industry, partly under the auspices of EEC, Nordic funds and the Danish Ministry of Energy and Danish Ministry of Industry. Work on high-temperature corrosion in conventional energy technology (3.4) is carried out within the European COST 501 project. This work is done in collaboration with a Danish firm and partly sponsored by the Ministry of Energy and The Ministry of Industry. In the area of ultrasonic testing the work is concentrated on quantitative ultrasonic examinations, especially the characterization of fibre reinforced materials. In the field of radiography standardization work is carried out within the neutron radiography working group sponsored by Euratom. Due to their proprietary nature, some of the projects in the technology programme are excluded from the progress report.

3.1. Fracture Testing

A round robin programme, sponsored by the Nordic Liaison Committee for Atomic Energy, on ductile fracture testing according to the J-integral based resistance curve technique, was completed. The programme, which involved a number of Scandinavian laboratories, displayed a high degree of scatter in the results and a detailed study of the data are under way. The mechanical tests carried out at Risø have been modelled with good results using the finite element programme ADINA.

Within the field of steels for offshore application, a number of CTOD tests have been carried out under the accreditation of the Danish National Testing Board. Furthermore the measurement of crack arrest properties sponsored by the Ministry of Industry

was continued and in a parallel effort, crack intensity factors associated with typical cracks in offshore structures were assessed.

3.2. Fatigue Testing Using Offshore Load Spectrum

An inter-Nordic program was started in January 1986. The aim of the project was to develop a fatigue testing procedure for simulating offshore loading conditions. The procedure is suitable for both crack growth tests and lifetime tests.

A log-linear load spectrum was defined based on a sequence of Rayleigh distributed load amplitudes with 5 different rms levels. The load sequence had a low-high-low rms sequence with 9 rms blocks. A simple algorithm was used to simulate the load sequence in laboratory tests. The resultant exceedance curve obtained during the tests closely followed the log-linear curve.

The procedure was implemented on two different computer aided test systems, namely PDP11 and Olivetti PC controlled servohydraulic test machines. The reliability of the testing procedures was verified by results from preliminary crack growth tests.

3.3. Brazing and Soldering

In order to decrease the consumption of silver in brazing alloys, an EEC sponsored project was continued and finished. The project aimed to determine the fatigue strength and creep properties of joints in copper tubes brazed with Cu-P alloys containing 0-15% Ag. The results obtained in low and high cycle fatigue and creep testing showed that the well known 40% AgCuZnCd brazing alloy could be substituted by the AgCu-P brazing alloys for the joining of oxygen-free copper materials.

Fig. 10. Structure of cast Cu - 8% P brazing alloy showing primary Cu and a coarse and fine eutecticum Cu-Cu_3P.

Contract work was continued on industrial applications of dip brazing and ultrasonic soldering of aluminium as well as vacuum brazing of stainless steels, aluminium alloys, superalloys and titanium.

3.4. High Temperature Materials

Studies of high temperature corrosion of commercial Fe- and Ni-base alloys in oxidizing/carburizing atmospheres relevant to the petrochemical industry were continued. The atmospheres, based on CH_4, CO, CO_2, H_2O and H_2, had at the test temperature a carbon activity of about unity and were oxidizing to Cr but reducing to Fe and Ni. Ni-base alloys with 16% Cr or less (e.g. Inconel 600) were very severely attacked at (700°-) 800°C, while Ni-base alloys with a higher Cr-content and all Fe-base alloys, even those with about 16% Cr, were attacked only to a very limited degree or were completely resistant. The mechanism of the attack on the Ni-base alloys and the complex structure of the corroded zone with carburization, internal oxidation and precipitation of free carbon were analysed.

Further, contract work on high temperature alloys (e.g. centrifugally cast HP and IN 519) included studies of the development of microstructure and mechanical properties after various heat treatments, and finally a number of failed or nearly failed components from industrial plants were examined and evaluated.

3.5. Fatigue Properties of Glass Fibre Reinforced Polyester

A conventional material for wingblades for windturbines is glass fibre reinforced polyester. An important property for the design life of the blades is their fatigue behaviour. Experiments were made on the glass/polyester material with fibre orientations of $\pm$ 5°, tested in tension-tension fatigue. The results indicated a fatigue limit, beyond 10^6 cycles, of about 0.5% strain. Further experiments on materials with fibre orientation 0° indicated a higher fatigue limit, and a project is in progress to study the fatigue behaviour of various glass/polyester materials with different fibre orientations. These are selected to simulate current materials used by the windturbine industry. In particular, very long time tests (approaching 10^8 cycles) are included.

The design limits (recommendations) derived for the glass/polyester material of $\pm$ 5° fibre orientation are 0.3% in tension fatigue and 0.2% in compression fatigue.

3.6. Ultrasonic Examiniation

Ceramic plates were examined with ultrasonic pulse echo technique. The aim was to detect small porosities of approx. 0.1 mm diameter. The plate thickness was more than 1 inch. The technique was optimized for finding pores throughout the plate and the best results were obtained with a 5 MHz focused transducer. By varying the water path the sensitivity could be focused in different depths of the plate.

In our computer-controlled scanning-system the three-dimensional position of a pore can be determined accurately. The resolution

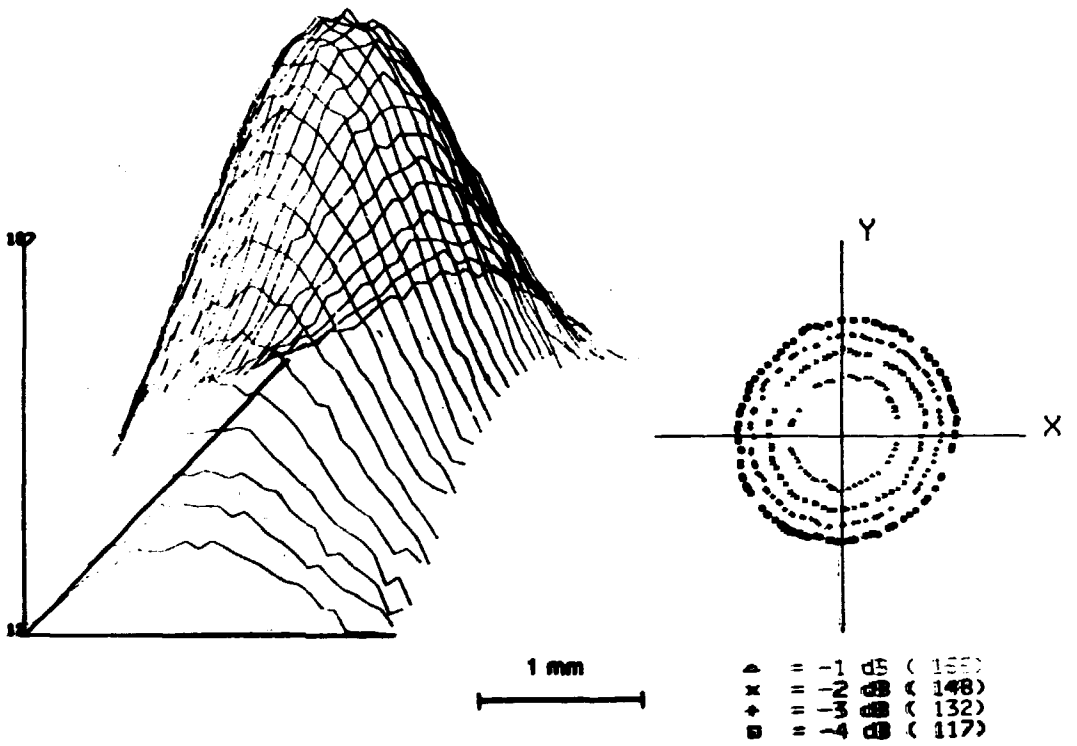

Fig. 11. Ultrasonic echo pulse from defect (0.2 mm diameter) in a ceramic plate, 15 mm from the surface. The transducer was moved in a rectangular scanning pattern. To the left the echo heights in the scanning lines (arbitrary units) are plotted. To the right curves for constant echo height are shown.

in the directions perpendicular to the measuring direction have been improved as shown in this table

Direction	Resolution until now	Resolution now
x	About 50 µm (Fixed)	Variable from 10 µm
y	Variable from 100±50 µm	Variable from 10 µm
Number of measurements pr. position	1	Several

The improvements have been achieved by changing the software and the hardware in the scanning system.

Work on different examination techniques for (carbon or glass) fibre-reinforced plates was continued. Sound velocity and RMS values determined by in-plane transmission was found to be unambiguously connected to the angle between fibre orientation and measuring direction. The calculated stress wave factors were also sensitive to defects and pores but the results were difficult to interpret.

The pulse echo reflector scanning technique can detect pores and other defects such as delaminations, and thus the two techniques supplement each other.

A license agreement on tube inspection, based on our patent, was renewed. Contract work on precise and fast tube inspection was carried out for the industry.

3.7. Neutron Radiography

Film density and dimensional measurements from 11 neutron radiography facilities participating in the Euratom Neutron Radiography Working Group (NRWG) Test Program were collected at Risø. At each NR facility 30 film/converter combinations were used. Nuclear beam components were calculated and radiographic quality was visually assessed from beam purity and sensitivity indicators. Absolute and percentage deviations of measurements made on neutron radiographs were calculated in relation to true dimensions of the calibration fuel pin. All the above data are listed and stored at Risø for further statistical evaluation. At the NRWG Sub-Group meeting it was agreed to make this evaluation by calculating standard deviations of measurements made at different NR-facilities for different film/converter combinations and different lengths of measurements.

4. CHEMICAL AND ELECTROCHEMICAL ENERGY RESEARCH AND DEVELOPMENT

The projects within this area include work on the storage of hydrogen as a metal hydride, work related to the use of batteries and work on the utilization of advanced ceramic materials. Many of the projects are sponsored by the Danish Ministry of Energy, some by other Danish and Nordic funds or by EEC. The results are published in the open literature and an index of the published works, the available experimental facilities and the scientific expert areas under the heading Chemical and Electrochemical Energy Research and Development is available on request.

4.1. Storage of Industrial Hydrogen

In a series of continuous cycling experiments magnesium powder with average particle diameter around 65 μm was exposed to hydrogen gases containing oxygen and nitrogen. The absorption and the desorption were measured over approx. 17 minutes each at a temperature of 375°C with small variations caused by the heat of reaction. The hydrogen gases used were N57 (99.9997%), H_2 + approx. 0.5% O_2 and H_2 + approx. 0.5% N_2. In pure hydrogen approx. 85% of the magnesium reacted within the absorption period to form MgH_2. When 0.5% O_2 or N_2 was added a prompt drop took place. When the Mg powder was exposed to 0.5% N_2 from the start, the uptake of hydrogen gradually rose to 35% over 50 cycles. The sample exposed to 0.5% O_2 suffered a permanent loss of approximately 20% of the initial capacity. The effect of 0.5% N_2 was similar but less pronounced.

4.2. Safety Aspects of Mg and MgH_2 Powders

A series of experiments relevant to the handling and storage of magnesium and its hydride was performed. With a comparable magnesium powder as reference 100-400 g hydride powder was ignited under various conditions and the temperature in and over the powder mass was recorded. Also the probable reaction of powder with water was investigated. The general conclusion was that magnesium hydride reacts in a way very similar to magnesium and should be handled accordingly, allowing though for the potential release of hydrogen during non-combusting heating.

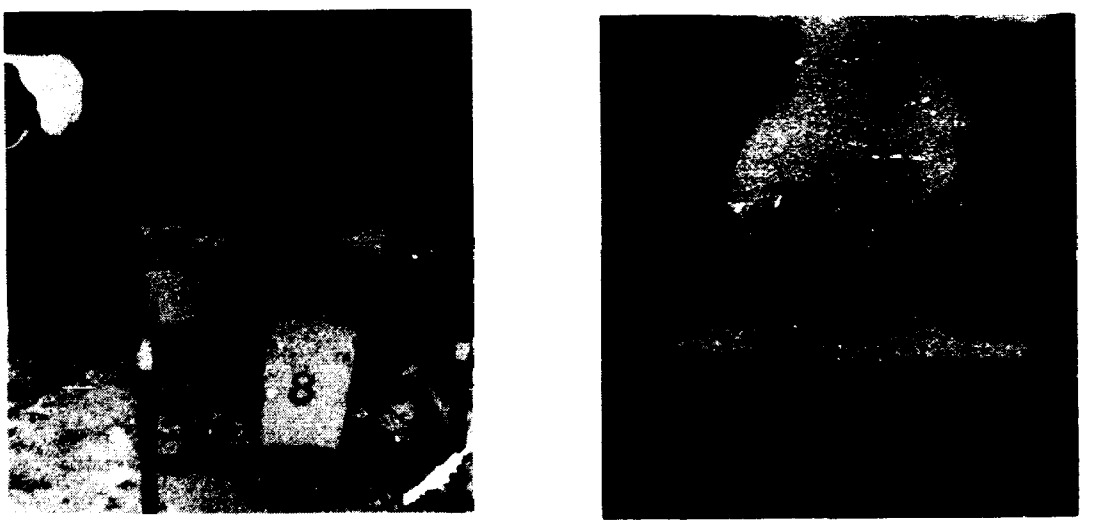

Fig. 12 a: Mg powder burns quietly in air if left alone. Two different powders are shown 10 minutes after ignition. b: Trying to extinguish with water has dramatic effects.

4.3. Development of a Hydride Based Fuel Cell

Work on fabrication of thin palladium membranes for electrode material was continued. Two routes were followed: one aiming at rolling the material to the desired thickness and one aiming at producing a thin supported membrane by utilizing RF-sputtering.

The rolling experiments resulted in sheets of thicknesses down to 18 µm. Between two rollings the material had to be annealed to approx. 1000°C. By one rolling we typically obtained a reduction in thickness of 20-50% depending on the initial sheet thickness. The rolled sheets were sufficiently strong to withstand pressure differences up to 3 bar when circular samples of diameter 25 mm were tested.

The sheets were electrochemically tested by potentiostatic measurements using a silver/silver chloride electrode as reference. The measurements were performed automatically, controlled by a computer and the results indicated that further reduction in thickness by 90-95% will increase the current to the anticipated current density.

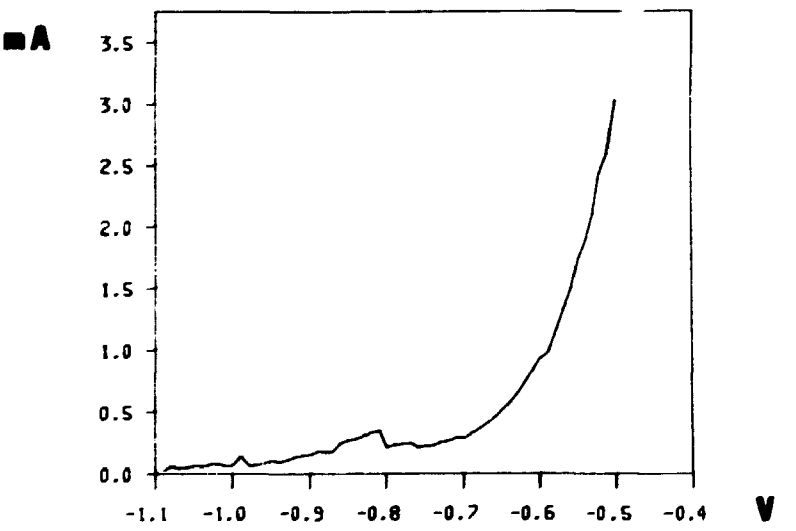

Fig. 11. Steady state current through the metal membrane as a function of membrane potential relative to a Ag/AgCl electrode. Membrane thickness and diameter: 18 µm and 25 mm respectively.

4.4. Materials Research for Fuel Cell Applications

Conductivity measurements of oxygen ion conductors at different temperatures and in various atmospheres have been performed using 2- and 4-electrode ac-impedance spectroscopy techniques. Computer programs facilitating the data reductions considerably have been developed, and correction models for the response of the measuring systems were studied. The materials studied were polycrystalline cerium-europium- and cerium-gadolinium oxides as well as single crystals and polycrystals of yttria stabilized zirconia (YSZ). As the ratio of bulk to grain boundary impedance of polycrystalline samples varies considerably, optical microscopy and electron microscopy studies were initiated to elucidate the relations between composition, microstructure and conductivity.

Studies of electrode materials such as platinum and cerium-praseodymium oxides were continued. A study of the oxygen reactions in a platinum/cerium-gadolinium oxide/platinum cell was performed by use of ac-impedance spectroscopy, steady state dc-polarization, and small amplitude cyclic voltametry methods on a two-electrode configuration. At temperatures above 700°C and at current densities up to 10 mA/cm^2 it was found that the oxygen electrode reaction is charge transfer controlled. The exchange current density for the reaction was determined as a function of both temperature and partial pressure of oxygen. For more comprehensive studies of the electrode-electrolyte interface characteristics special three-electrode configurations have been prepared by use of rf-sputtering thin film techniques, and a sample holder for three-electrode ac-impedance and voltametric measurements have been developed. The production method and the thermal stability of porous platinum electrodes have been studied. Scanning electron microscopy has been used to record the agglomeration kinetics of thin rf-sputtered platinum and platinum-palladium films. Single- and polycrystalline YSZ, aluminium oxide and magnesium oxide were used as substrates. Effects of temperature, time, film thickness, alloying, surface impurities, and substrate morphology have been considered. Transmission electron microscopy and X-ray diffraction were used to show that the agglomer-

Fig. 14. Agglomeration of a 2000 A sputtered Pt-film on a {100} surface of yttria-stabilized zirconia.

ation of thin platinum films on a {100} surface of single crystals of YSZ leads to epitaxial platinum layers having the [111] direction normal to the YSZ surface. In the surface plane the platinum [110] direction is parallel to one of the YSZ <100> directions.

The defect structure of YSZ has been studied by quasielastic diffuse neutron scattering on single crystals in collaboration with the Physics Department, Risø, Harwell and Clarendon Laboratory, UK. Measurements on six samples with 9.4 to 24 mole % yttria in zirconia suggest a structural model with two defect components. One component, dominant at low yttria doping levels, is a tetrahedral distortion of the cubic fluorite type YSZ lattice, consistent with the room temperature zirconia structure. For the larger yttria concentrations, defect components with a tendency towards formation of the $Zr_3Y_4O_{12}$ structure prevail. The dynamical changes of the defect configurations have been studied, by measurements of the q- and temperature dependence of the quasielestic linewidth, in order to clarify the mechanisms of the ionic conductivity in these oxygen conducting materials.

4.5. Thin Film Techniques by RF-Sputtering

Modern rf-sputter systems for thin film technology have a great flexibility. A large number of materials, including metals, semiconductors, glasses and ceramics may be deposited on almost any solid surface. Surface cleaning and microlithography in thin films are also possible by etching processes. An rf-sputter system, which focuses on fabrication of materials for batteries, fuel cells and sensors, has been developed. As handling in protective gases is required for many of these materials, the system has been equipped with a glove box. A laminar flow bench and clean water rinse systems are available for preparation of substrates. Microlithography processes are performed by use of photoresist techniques in combination with chemical etching and sputter etching. Methods for fabrication and bonding of target materials for thin film sputter processes, especially ceramic target materials, have been developed. Characterization of thin

Fig. 15. Sputtering equipment with glove box facility.

films are carried out by scanning electron microscopy, transmission electron microscopy, optical microscopy, X-ray diffraction and electrical methods.

4.6. Thin Solid Electrolyte Layers on Li

The study of solid electrolyte layers on Li electrodes in $SOCl_2$, SO_2Cl_2 and SO_3 solutions was continued within the frame of a new project. The purpose of this project was two-fold.

Firstly, the kinetics of the formation of the layer on Li-electrodes exposed to the solutions were investigated by SEM and impedance spectroscopy. For Li in $SOCl_2$ the results so far indicate that nucleation and growth of LiCl crystals is the rate determining step in the early stages of the film formation.

Secondly, the properties (i.e. ionic and electronic conductivity) of films formed in solution were compared to the properties of films made by ac-sputtering and by vapor deposition. The development of these two techniques has been an important part of the project during its first year.

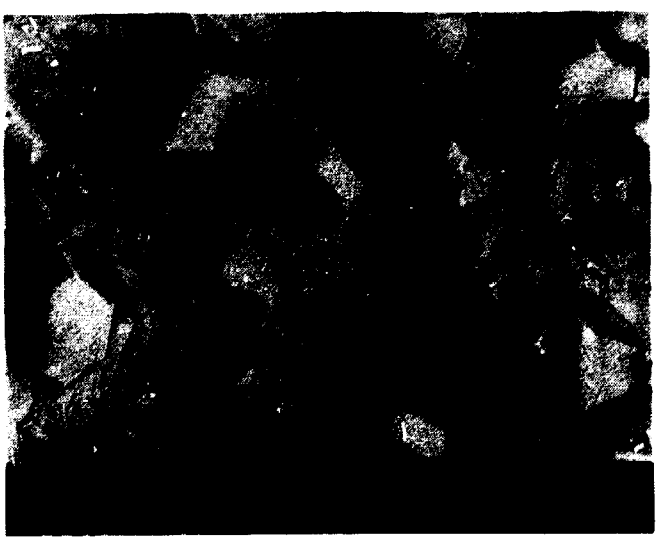

Fig. 16. SEM micrograph of LiCl crystals on a Li electrode after exposure to 1.8 M $LiAlCl_4$ in $SOCl_2$ for one year.

4.7. Materials Research on Advanced Ceramic Materials

Advanced ceramic materials, i.e. ceramics with improved mechanical strength, fracture toughness and thermal stress resistance (structural ceramics), have been developed in two research projects: a Danish national project in close collaboration with the Technical University, Copenhagen, and the Technological Institute, also in Copenhagen, and in a Nordic project in collaboration with the Technological Institute of Iceland (Reykjavik, Iceland) and ASEA Cerama (Robertsfors, Sweden). In the Danish national project the main efforts were directed towards development of methods for measurements of mechanical properties (fracture strength and fracture toughness) as well as characterization of the mechanical properties of ceramic specimens prepared from commercial powders. The materials considered in this project were zirconia toughened oxides such as YPSZ (yttria partially stabilized zirconia, TZP (tetragonal zirconia polycrystal) and ZrO_2 - 20 wt% Al_2O_3. Techniques for preparation of ceramic specimens by pressing (uniaxial and isostatic) followed by sintering were also developed in this project. Finally techniques for preparation of zirconia toughened mullite from cheap raw materials were also investigated in a licentiate project in collaboration with the Technical University, Denmark. The purpose of the Nordic project is to develop hydrothermal processes for production of oxide powders with good pressing and sintering properties. The materials considered in this project were YPSZ and Gd_2O_3 doped CeO_2 (oxygen conductor). The work at Risø was directed towards preparation of specimens from powders prepared hydrothermally in Iceland (by pressing and sintering) and towards characterization of the mechanical and electrical properties of these specimens. For YPSZ it was concluded that powders produced by the hydrothermal methods have properties similar to commercially available powders. The hydrothermal technique thus seems to be a promising technique for preparation of starting powders for advanced ceramic materials.

4.8. Development and Testing of Oxygen Sensors

Development and testing of oxygen sensors based on solid oxide electrolytes were carried out in collaboration with Dansensor System A/S in a project supported by the Danish Ministry of Energy. The main work was directed towards testing of sensors under extreme conditions, i.e. high temperature (up to 1450°C) and very low oxygen pressures. Two types of oxygen sensors were tested: a zirconia sensor based on YSZ (yttria fully stabilized zirconia) and a thoria sensor based on YDT (yttria doped thoria). Both types were found to be useful up to 1450°C but about five orders of magnitude lower oxygen pressure can be measured with the thoria sensor.

5. FUEL ELEMENTS

The Danish water reactor fuels programme has continued to utilize the irradiation facilities in the DR 3 materials testing reactor at Risø and the OECD Halden Reactor in Norway. Extensive post-irradiation examinations were performed in the Risø hot cells. The internationally sponsored three-year project, "The Risø Transient Fission Gas Release Project" has now been completed. This was a follow-on programme to the first "Risø Fission Gas Project" that was executed in 1980-1981. Preparations are well under way for a new project "The Third Risø Fission Gas Project". This project will utilize a new Risø technique to instrument fuel segments (previously irradiated in a power or a test reactor) with fresh thermocouples, thus enabling accurate, direct measurements of the fuel temperature during transient tests with high-burnup fuel.

Additional information on fuel performance becomes available from international collaboration arrangements, i.e. the OECD Halden Reactor Project (Norway), the Super-Ramp Extension project at Studsvik (Sweden) and Battelle's "High Burnup Effects Program" (USA).

In previous years, the EEC has sponsored extensive examinations of plutonium-enriched fuels from light-water reactors, and an important part of this work was performed in the Risø hot cells. The results from all these examinations have now been "synthesized" in a joint evaluation, to demonstrate the feasibility of plutonium recycling in light-water reactors.

5.1. UO_2-Zr Irradiations

The irradiation in DR 3 of standard fuel pins was continued. The maximum burnup levels of the current irradiations are 54000 and 47000 MWD/tU for BWR and PWR type fuel, respectively. The ir-

radiation facilities were also used for the performance testing of LOWI duplex fuel (max. burnup 54000 MWD/tU, see also previous progress reports) and for the prototype testing of previously irradiated fuel that has been instrumented in the Risø hot cells with a central fuel thermocouple.

The three Danish test fuel elements in the Halden reactor have now reached the following estimated burnups (average assembly, after correction for depletion):

IFA No.	165	201	202
MWD/tU	44,700	45,200	42,100

5.2. The Risø Fission Gas Projects

The now completed, second Risø project, "The Risø Transient Fission Gas Release Project", had the objective to study the kinetics of fission gas release during power transients with high-burnup fuel. The experimental program comprised 17 transient tests (15 tests refabricated with pressure transducers and 2 tests with unopened fuel pins for comparison) with extensive hot cell examinations. Important results were obtained with various combinations of the following test parameters: Transient power histories (levels up to 500 W/cm), burnup (15,000-50,000 MWD/tU, fill gas (Xe, He at various pressures) and gap size (1.5 and 2% of the pellet diameter). This work has now been evaluated in a final project report.

Besides the continuous pressure monitoring in the second project, there is a strong interest in direct measurements of the fuel temperature during the transient tests with high-burnup fuel. So far, this has not been possible using fuel irradiated in a power reactor. Risø therefore started the development of a technique to instrument fuel segments (previously irradiated in a power or a test reactor) with fresh thermocouples. In-pile proto-

type testing has been performed successfully. Important use of this technique will be made in "The Third Risø Fission Gas Project", a new, internationally sponsored four-year project.

6. PARTICIPATION IN INTERNATIONAL COLLABORATION

The department is engaged in the following types of international collaboration: joint scientific and technical projects, committee work, reception of research fellows, and technical and scientific meetings.

The department was represented in the following committees:

The Information Exchange Group under the European Space Agency on Carbon Fibre Reinforced Plastics,

The Halden Programme Group,

The IAEA International Working Groups on "Reliability of Reactor Pressure Components" and "Water Reactor Fuel Performance and Technology" (Chairmanship),

The Super-Ramp Extension Project Committee, The Project Committee of the Battelle High Burnup Performance Programme (HBEP), The Principal Working Group No. 3 (Primary Circuit Integrity) of the NEA Committee on The Safety of Nuclear Installations (CSNI), The EEC Working Group on "Reference Materials for the Elemental Analysis of Plutonium",

The COST 501 Management Committee on Materials for Energy Conversion Using Fossile Fuels,

The European Coal and Steel Community, Executive Committee No. 5: Failure Mechanisms and Design,

The Euratom Neutron Radiography Working Group,

The Council of the International Confederation of Thermal Analysis,

The Nordic Committee for Thermal Analysis (Chairmanship),

The Technical Commission of the International Institute of Welding, Commission I, "Gas Welding and Allied Processes", Subcommission A, "Brazing and Surfacing",

The Fusion Technology Steering Committee,

The Fusion Materials Expert Groups of the European Fusion Technology Programme: "Structural Materials" and "Breeding Materials",

The Editorial Board of "Composites Science and Technology", "Journal of Materials Education", "Thermochimica Acta", "Microstructure and Texture" and "Journal of Nuclear Materials".

7. EDUCATION AND TRAINING

N. Hansen and K. Rørbo gave regular lectures on materials science to students at the Danish Academy of Engineering. J.B. Bilde-Sørensen, N. Hansen, T. Leffers and H. Lilholt acted as external examiners at examinations for the Technical University of Denmark, O. Toft Sørensen acted as external examiner at examinations for the Technical University of Norway and O. Bøcker Pedersen acted as external examiner for the degree of dr.ing. at the Technical University of Norway.

Post-Graduate Projects

Two post-graduate students, one from the Technical University of Denmark, the other from Aalborg University Centre, worked in the department on the following projects in preparation for their licentiate (Ph.D.) theses:

P.L. Husum: Fremstilling og karakterisering af avanceret teknisk keramik. (The Fabrication and Characterization of Advanced Technical Ceramic Ma-

T. Lorentzen: Måling af indre spændinger med neutrondiffraction (Measurement of Internal/Residual Stresses by Neutron Diffraction).
terials).

Two post-graduate students, one from the Department of Metallurgy and Materials Science, Cambridge University, the other from Institute of Materials, University College, Cardiff, worked part time in the department on the following projects in preparation for their Ph.D. theses:

Ph. Withers: Interfaces and Internal Stresses in Metal Fiber Composites.

I. Barker: Development of Techniques to Measure Local Misorientations and Application of Such Techniques in the Study of Deformed and Partly Recrystallized Structures in Metals.

Degrees conferred

The Technical University of Denmark conferred the degree of lic. techn. (Ph.D.) on S.E. Soliman, who left the department in the middle of 1986.

PUBLICATIONS

A Study of the Disorder in Heavily Doped $Ba_{1-x}La_xF_{2+x}$ by Neutron Scattering, Ionic Conductivity and Specific Heat Measurements.

N.H. Andersen, K. Clausen, J.K. Kjems and J. Schoonmann, J. Phys. C.: Solid State Physics 19, (1986) 2377-2389.

The ionic disorder in single crystals of the fluorite-type solid solutions $Ba_{1-x}La_xF_{2+x}$ (with x=0.209 and x=0.492) has been studied in the temperature range from room temperature to 800°C by diffuse neutron scattering, ionic conductivity, and specific heat measurements. From the diffuse neutron scattering it was found that the disorder was dominated by 222 clusters, which at low temperatures (T<400°C) were ordered along the [100] direction in aggregates of up to four 222 clusters. The correlation between the different 222 clusters in the aggregates is slowly lost when entering into the fast ion phase. The lifetime of the 222 clusters could not, even at the highest temperatures, be determined by neutron scattering ($\tau >> 10^{-10}$s), in agreement with NMR results which suggest a jump frequency below 75 MHz. The temperatures at which the steepest slopes are found in the loss of correlations and in the conductivity coincide at approximately 650°C. At this temperature no clear anomaly is observed in the specific heat. Based on these findings we propose a conduction mechanism where F^- ions are moving through the lattice by means of rearrangements of the 222 clusters.

Diffuse Neutron Scattering Study of $Cu_{2-x}Se$.

R.J. Cava, N.H. Andersen and K. Clausen, Solid State Ionics 18 & 19 (1986) 1184-1187.

We have measured the diffuse neutron scattering in the hkk plane for Cu_2Se and $Cu_{1.8}Se$ at 180°C and 51°C, respectively, in the cubic antifluorite type phase. The diffuse scattering shows significant structure, indicative of correlated short range mobile ion ordering. The short range order is found to be dependent on the concentration of vacant mobile ion sites.

Neutron Diffraction and TSDC on $Ba_{1-x}U_xF_{2+2x}$ Solid Electrolytes.

M. Ouwerkerk, N.H. Andersen, F.F. Veldkamp and J. Schoonmann, Solid State Ionics 18 & 19 (1986) 916-921.

The defect structure of fluorite-type $Ba_{1-x}U_xF_{2+2x}$ solid solutions, which exhibit fast fluoride ion conductivity, has been investigated by quasi-elastic diffuse neutron scattering (QDNS) experiments, and thermally stimulated depolarisation current (TSDC) measurements. A comparison with model calculations reveals (2:2) clusters to dominate the defect structure. From 10 to 400K TSDC spectra reveal for $x < 0.05$ six relaxation peaks of dipolar nature, along with the relaxation of macroscopic space charge. Conductivity parameters calculated from the latter relaxation compare very well with those obtained from a.c. ionic conductivity studies. The low-temperature relaxation at 29.5K is ascribed to depolarisation steps of (212) clusters. Evidence for the relaxation of a minor amount of $(U_{Ba}F_i)^x$ dipoles is found. A direct indication of the freedom of orientation of a (212) cluster is obtained from an initial QDNS-study on a polarised crystal.

Neutron Scattering Investigation of the Defect Structure of Y_2O_3-Stabilised ZrO_2 and Its Dynamical Behaviour at High Temperatures.

R. Osborn, N.H. Andersen, K. Clausen, M.A. Hackett, W. Hayes, M.T. Hutchings and J.E. MacDonald, Mater. Sci Forum 7 (1986) 55-62.

Yttria-stabilised zirconia has many uses as an oxygen-ion conductor, but despite the attention of several groups of workers over recent years the exact conduction mechanism is not yet fully understood. Recent theoretical work has suggested that in the case of calcia-stabilised zirconia the formation of microdomains of a mixed oxide is as equally likely to occur as a uniform dispersal of dopant ions. In order to investigate the static and dynamic behaviour of the defect structure, coherent diffuse neutron scattering from single crystal samples of 9.4, 12, 15, 18 mol % Y_2O_3, has been investigated. The measurements were made using triple-axis spectrometers. At 293K the diffuse scattering in the $(1\bar{1}0)$ plane shows a characteristic distribution of intensity with a scattering vector which changes systematically as the dopant level is increased. The temperature variation of the scattering from the 9.4 mol% sample has been investigated to 1900°C and a complete distribution of intensity in the $(1\bar{1}0)$ plane measured. At the highest temperatures the scattering becomes partly quasielastic, although the integrated intensity retains the same general distribution in reciprocal space. The areas of most intense scattering in reciprocal space appear to have two components at high temperature, one elastic and one quasielastic, and a preliminary measurement of the variation with wave vector has been made.

Risøs metoder til studier af faststofelektrolyt'er (The Methods at Risø for Studies of Solid State Electrolytes).

N.H. Andersen, J.J. Bentzen, M. Mogensen, F.W. Poulsen and O.T. Sørensen, In: Generel og anvendt elektrokemi i Danmark (General and Applied Electrochemistry in Denmark). Extended abstracts of the symposium Generel og anvendt elektrokemi i Danmark, Danmarks tekniske Højskole, 9 April 1986. (Dansk Elektrokemisk Forening, Selskabet for Analytisk Kemi, Lyngby, 1986) 16-17

The Defect Structure of Yttria-Stabilized Zirconia, Studied by Quasielastic Diffuse Neutron Scattering.

N.H. Andersen, K. Clausen, M.A. Hackett, W. Hayes, M.T. Hutchings, J.E. MacDonald and R. Osborne, Physica 136B (1986) 315-317.

The static defect structure of the oxygen ion conductor Y_2O_3 stabilized zirconia has been studied at room temperature by coherent diffuse neutron scattering from single crystal samples containing nominally 9.4, 12, 15 and 18 mol% Y_2O_3. There are two principal contributions to the observed diffuse intensity. The first arises from tetrahedral distortions in small vacancy free regions of the crystal which decrease in volume as the dopant level increases. The second arises from correlated vacancies and their associated relaxed ions in the remainder of the crystal. The 9.4 mol% sample has been studied at elevated temperatures. The scattering becomes partly quasielastic, but the correlations persist to the highest temperatures studied (1900°C). The temperature- and Q-dependence of the energy width has been studied at selected positions in reciprocal space.

Tyndfilmteknik på Risø (Thin Film Techniques at Risø).

N.H. Andersen, Risønyt No. 3 (1986) 3.

Application of Resistance Foil Strain Gauges on Fibre Reinforced Materials.

S.I. Andersen, In: Mechanical Characterization of Fibre Composite Materials. Proceedings of the Conference on Mechanical Characterization of Fibre Composite Materials, Ålborg Universitet 3-4 June 1986. Edited by R. Pyrz (Ålborg Universitet, Ålborg, 1986) 94-111.

Resistance foil strain gauges are among the main measuring sensors used by many scientists, research engineers and design engineers working with materials science and structural design problems. In the paper it is shwon, that it is necessary to correct for the difference in transverse sensitivity between the gauges and the material, and that this correction can be substantial, depending on the degree of anisotropy of the material. Further, misalignment of gauges may introduce errors, the magnitude of which also is dependent of the degree of anisotropy. The correction for the difference in transverse sensitivity implies that it is necessary always to measure in two directions at each gauge position and to use gauges with known transverse sensitivity coefficients. The correction is simple and should be done by routine in the dataprocessing, during or after the measurement. Misalignment of gauges may introduce serious errors, but as they are dependent of both the misalignment angle relative to the intended position and of the orientation relative to the principal material directions, correction is not straightforward. The error may be considered as an uncertainty in the measurement, and it has to be minimized. The errors are most pronounced at unidirectional laminates, often used for material characterisation in the laboratory, and the error has a maximum at the fibre angle often used in measurement of shear properties.

Udvikling af iltsensorer på Risø. (The Development of Oxygen Sensors at Risø).

J.J. Bentzen and O. Toft Sørensen, In: Generel og anvendt elektrokemi i Danmark. Extended abstracts of the symposium Generel og anvendt elektrokemi i Danmark, Danmarks Tekniske Højskole, 9 April 1986. (Dansk Elektrokemisk Forening og Selskabet for Analytisk Kemi, Lyngby, 1986) 35-36.

Deformation Bands in <120> Grains in Coarse-Grained Aluminium.

J.B. Bilde-Sørensen, Mater. Sci. Eng. 81, (1986) 211-216. Also published in Low-Energy Dislocation Structures. Proceedings of the International Conference on Low-Energy Dislocation Structures, Charlottesville, 10-14 August 1986. Edited by M.N. Bassim et al. (Elsevier Sequoia, Lausanne, 1986) 211-216.

Coarse-grained aluminium, deformed in tension to a strain of 0.05, was examined in a scanning electron microscope by channelling contrast. Pronounced bands with a width typically of the order of 200 μm were found in some grains with an orientation close to [120]. When observed on surfaces close to [001], the boundaries between the bands were parallel to [010] and the neighbouring bands were rotated around [100] with respect to one another.

Two slip systems in a critical relationship are equally stressed with a Schmid factor of 0.49 in grains with a [120] orientation, namely (a/2)[01$\bar{1}$](111) and (a/2)[011](11$\bar{1}$). The Schmid factor for the highest stressed secondary systems has a local minimum of 0.245 at [120]. The application of Frank's equation shows that the only boundaries without long-range stresses that can be formed by combination of the two sets of dislocations, (a/2)[01$\bar{1}$](111) and (a/2)-[011](11$\bar{1}$), allowing for glide only, are tilt boundaries with a normal [001] and a rotation axis along [1$\bar{1}$0] and twist boundaries with a normal [100] and rotation axis along [100]. A consideration of the boundary energies indicates that the twist boundary has a lower energy than the tilt boundary for a given amount of deformation. The observed structure thus agrees with that expected on the basis of a low energy structure argument.

Grundstofanalyse i mikrometerskala (Elemental Analysis at the Micrometer Level).

J.B. Bilde-Sørensen, Risønyt No. 1 (1986) 3.

A description is given of the windowless energy-dispersive X-ray detector which has been installed on the scanning electron microscope at Risø's Metallurgy Department. The windowless detector has the advantage over common detectors that also light elements as carbon, nitrogen and oxygen can be detected. Examples of application of the technique are included.

Fatigue Properties of Glass Fiber Reinforced Plastics.

P. Brøndsted, In: Materials Science. Proceedings of the Fourth Scandinavian Symposium on Materials Science, Trondheim, 25-26 August 1986. Edited by C. Thaulow et al. (The University of Trondheim, Trondheim, 1986). 97-107.

The increasing use of fiber reinforced plastics especially as critical carrying parts in constructions has lead to development of test methods for measuring the mechanical properties. Because of the anisotropy of these materials, standard test methods cannot be used and especially new failure criteria have to be considered. In connection with the Danish wind turbine program these materials were and still are used in the carrying beams in the wings and a test program for measuring the fatigue properties of these materials has been and is still running in the Metallurgy Department at Risø National Laboratory.

In this paper a theoretical overview for the fatigue properties of these materials are given. A failure criterion based on observed changes in initial stiffness is proposed and a new developed experimental procedure based on an online computerized testing system is described. The materials tested are glass fiber reinforced polyester having a volume fraction of about 50% and a +/- 5° orientation. Because of the extreme ratios betwen the tensile/com-

pression strengths and the shear strengths it has been necessary to develop a special specimen geometry and a special gripping fixture to avoid failure in the specimen gripping area. The test results presented in this paper are based on constant load amplitude fatigue test with an R-value of 0.1 and the results are presented as S-N curves.

Udmattelsesprøvning af glasfiber/polyester kompositmaterialer. (Patigue Testing of Glass Fiber/Polyester Composite Materials).

P. Brøndsted, In: Måling af materialeegenskaber. (Measurement of Materials Properties). Dansk Metallurgisk Selskabs Vintermøde, Risø, 6-8 January 1986. Edited by H. Lilholt and G. Skjelsager. (Dansk Metallurgisk Selskab, Lyngby, 1986) 21-35.

Same abstract as above.

Brazed Joints in High Temperature Materials.

J. Christensen, In: High Temperature Materials. Proceedings of the Denmark-Israel Binational Symposium on High Temperature Materials, Jerusalem, 14-15 April 1986. Edited by A. Kaufman. (Ministry of Science and Development, National Council for Research and Development, Jerusalem, 1986) 15 pp.

Brazed joints are being increasingly used to manufacture assemblies for high temperature applications. Although much information has been published on the properties of brazed joints, the data are frequently related to a specific application; thus, there is a shortage of general information on the basic properties of brazed joints made in high temperature materials. This paper reviews their characteristics, discussing the effect of the brazing cycle, the parent metal/filler reactions, joint design and heat treatment on the properties of the brazed component. Quality control procedures can be applied with confidence and make the high temperature brazing process one of the most reproducible of all the joining processes.

Fatigue and Creep Prcperties of CuP-Brazed Joints and of the Brazing Alloys.

J. Christensen and P. Brøndsted, Risø-M-2618 (Risø National Laboratory, Roskilde, 1986) 43 pp.

Mechanical creep and fatigue properties of copper joints brazed with various AgCuP-brazing alloys have been investigated. Also the fatigue strength of the as cast brazing alloys have been measured since they are often present as a continuous phase in the gaps, and thus will be determining for the properties of the brazed joints. The creep and fatigue strength measured showed that the strengths of the joints brazed with the various brazing alloys as well as the strength of the alloys themselves were all well above the strength of copper heat treated according to standard brazing procedures. Results of direct use for practice were demonstrated by the fatigue tests carried out on standard brazed joints of normed copper tubes. It was found that brazed joints with irregularities and overlap lengths of only 30% of the normed always fractured in the copper tube. With respect to the creep and fatigue properties measured the normally used 30-50% Ag-bearing brazing alloys can be substituted by the 0-15% AgCuP alloys. Thereby the silver consumption can be reduced and economical savings obtained by industry.

Måling af brudsejhed ved stop af hurtig revneudbredelse. (The Measurement of Steel Fracture Toughness at Fast Crack Arrest).

C.P. Debel, In: Måling af materialeegenskaber. (Measurement of Materials Properties). Dansk Metallurgisk Selskabs Vintermøde, Roskilde, 6-8 January 1986. Edited by H. Lilholt and G. Skjelsager. (Dansk Metallurgisk Selskab, Lyngby, 1986) 85-94.

The determination of a materials fracture toughness at the moment of crack arrest is a difficult task since the conditions acting at arrest have disappeared only milliseconds later, and since a subsequent static evaluation may be in error. At present the most suitable method of measuring the crack arrest toughness appears to be one based on the use of specimens designed to suppress dynamic effects.

Activities and Achievements of the Euratom Neutron Radiography Working Group.

J.C. Domanus, Mater. Eval. 44 (1986) 114-119.

Various activities of the Euratom Neutron Radiography Working Group (NRWG) and its subgroups are described. This working group, created in 1979, holds its annual meetings each year at a different neutron radiography (NR) center of the European Community. The NRWG has published a handbook on NR and recently reference neutron radiographs of nuclear reactor fuel. For the determination of neutron beam components, image quality, and accuracy of dimensional measurements from neutron radiographs, special indicators and calibration fuel pins were produced and are now being tested under a NRWG test program. Optimum conditions for use of nitrocellulose film are also investigated. The International Neutron Radiography Newsletter informs all concerned about activities in this field.

Assessment of Radiographic Image Quality by Visual Examination of Neutron Radiographs of the Calibration Fuel Pin.

J.C. Domanus, Risø-M-2578 (Risø National Laboratory, Roskilde, 1986) 13 pp.

Up til now no reliable radiographic image quality standards exist for neutron radiography of nuclear reactor fuel. Under the Euratom Neutron Radiography Working Group (NRWG) Test Program neutron radiographs were produced at different neutron radiography facilities within the European Community of a calibration fuel pin. The radiographs were made by the direct, transfer and track-etch methods using different film recording materials. These neutron radiographs of the calibration fuel pin were used for the assessment of radiographic image quality. This was done by visual examination of the radiographs and assessing their radiographic image quality on an arbitrary scale.

Can Neutron Beam Components and Radiographic Image Quality be Determined by the Use of Beam Purity and Sensitive Indicators?

J.C. Domanus, Risø-M-2579 (Risø National Laboratory, Roskilde, 1986) 18 pp.

In the Euratom Neutron Radiography Working Group Test Program beam purity and sensitivity indicators, as prescribed by the ASTM E 545-81, were used together with the NRWG beam purity indicator-fuel and calibration fuel pin. They were radiographed together at neutron radiography facilities of the European Community. The direct, transfer and track-etch methods using different film recording materials were used. Neutron beam components were calculated from film density measurements under the beam purity indicators, and radiographic image quality was assessed by visual examination of the sensitivity indicator. Results obtained under the NRWG Test Program are summarized and compared.

Euratom Neutron Radiography Working Group.

J.C. Domanus, Risø-M-2576 (Risø National Laboratory, Roskilde, 1986) 17 pp.

In 1979 a Neutron Radiography Working Group (NRWG) was constituted within Euratom with the participation of all centers within the European Community where neutron facilities were available. The main purpose of NRWG was to standardize methods and procedures used in neutron radiography of nuclear reactor fuel as well as establish standards for radiographic image quality of neutron radiographs. The NRWG meets once a year in each of the neutron radiography centers to review the progress made and draw plans for the future. Besides, ad-hoc sub-groups on different topics within the field of neutron radiography are constituted. This paper reviews the activities and achievements of the NRWG and its sub-groups.

International Neutron Radiography Newsletter.

J.C. Domanus, Risø-M-2577 (Risø National Laboratory, Roskilde, 1986) 8 pp.

At the First World Conference on Neutron Radiography it was decided to continue the "Neutron Radiography Newsletter", (INRNL), with J.C. Domanus as editor. The British Journal of Non-Destructive Testing (BJNDT) has agreed to publish the INRNL in its column "NDT Bookcase". The Revue Practique de Control Industriel has also agreed to publish the French version of the INRNL. Up till now 12 issues of the INRNL were published in the BJNDT. They are reviewed in the report.

Reference Neutron Radiographs of Nuclear Reactor Fuel.

J.C. Domanus, Risø-M-2575 (Risø National Laboratory, Roskilde, 1986) 12 pp.

Reference neutron radiographs of nuclear reactor fuel were produced by the Euratom Neutron Radiography Working Grou, and published in 1984 by the Reidel Publishing Company. In this collection a classification is given of the various neutron radiographic findings, that can occur in different parts of pelletized, annular and vibro-compacted nuclear fuel pins. Those parts of the pins are shown where the appearance differ from those for the parts as fabricated. Also radiographs of these as fabricated parts are included. The collection contains 158 neutron radiographs, reproduced on photographic paper (twice enlarged) and on duplicating film (original size).

A Positron Annihilation Study of Hydrated DNA.

J.M. Warman and M. Eldrup, Biopolymers 25 (1986) 1865-1874.

Positron annihilation measurements are reported for hydrated DNA as a function of water content and as a function of temperature (20 to -180°C) for samples containing 10 and 50% wt of water. The ortho-positronium mean lifetime and its intensity show distinct variations with the degree of hydration and with temperature for the 50% sample. The 10% water sample was relatively insensitive to temperature variation. The results indicate that hydrated DNA containing up to 10% water behaves as a rigid crystalline solid but that the rigidity markedly decreases with a further increase in water content until, for approximately 50% water, its properties resemble more those of a highly viscous fluid.

Positron Annihilation in Pivalic Acid. Temperature Dependence of Angular Correlation Curves.

P.C. Jain, M. Eldrup, N.J. Pedersen and J.N. Sherwood, Chem. Phys. 106 (1986) 303-313.

Positron annihilation angular correlation curves have been measured as a function of temperature for trimethylacetic (pivalic) acid in both the brittle and plastic phases. A simple fitting of the data to a sum of three gaussians shows the presence of a narrow component due to para-positronium (p-Ps) annihilation. In the brittle phase the intensity of the narrow component is inconsistent with previous positron lifetime data. A more detailed analysis, requiring consistency with the lifetime data, results in the determination of the shapes of the angular correlation components for free positron-, pick-off- and p-Ps intrinsic annihilation. The p-Ps component has a width (fwhm) of 3.75 mrad in the brittle phase, probably due to Ps self-trapping or trapping in defects smaller than molecular vacancies. In the plastic phase the width (fwhm) is 3.25 mrad which is ascribed to Ps localization in vacancies and divacancies in accordance with positron lifetime data.

Anvendelse af en ultralydmetode til kvalitetskontrol og levetidsberegninger for kompositmaterialer. (The Utilization of an Ultrasound Method for Quality Control and Calculations of Life Times of Composite Materials).

H.E. Gundtoft, H. Jensen and T. Nielsen, rapport nr. S-8611/205-29-85 til Teknologirådet (Risø National Laboratory, Roskilde, 1986) 75 pp.

The report treats:

- measurements with pulse-echo technique (Through Transmission) with scanning and improvement of this technique.
- finishing of in-plane transmission technique so that it can measure in a reproducible, quick and mobile way.
- comparison of results from the two techniques on different subjects (different thicknesses and different manufacturing technologies.

Through transmission reflector technique is reasonably robust and besides sensitive to e.g. manufacturing failures, which give increased porosity content in the sheets. The results show that it is very important with vacuum during manufacturing to get low porosity (and good strength). The pulse-echo technique can give several informations about defects in the material, but this method can be too sensitive to discover the severe defects.

The in-plane transmission method has been developed to a level, where it gives reasonable results for sheets, where no defects are found by scanning. In a sheet where scanning with reflector technique gives many indications, the scattering on "Stress Wave Factors" is big. The velocity- and RMS-measuring is, however, robust against this type of defects. However, the vel-

ocity determination is sensitive to electric noise coming from the Main Bang. This could be improved by using separate transmitter and receiver. The conclusions must be, that the method functions satisfactory for some of the factors. Other factors however, are too sensitive to variations in the manufacturing technique.

Automatic Quantitative Non-Destructive Examination in Computerized Scanning.

H.E. Gundtoft, In: Automated Non-destructive Testing. Proceedings of a Topical Seminar, University of Idaho, Idaho Falls, 28-30 June 1983. Edited by W.J. McGonnagle. (Gordon and Breach Science Publishers, New York, 1986) 111-125.

At Risø National Laboratory we have studied the problems around sensing and documentation of "defects" for several years, especially with regard to ultrasonic and radiographic non-destructive examinations. Our approach has been to work with automatic or semiautomatic systems and try to get reproducible and accurate results. Thus we try to exclude the influence of manual operation and of the operator on the examination. Naturally the set-up and calibration of the system prior to the examination is of vital importance for the results, so here we need a qualified operator. This paper deals with equipment and techniques that we have developed and used at Risø, and some examples of problems solved by us. In all our work we make an automatic scanning movement and store the results together with the measuring position. The scanning system and measuring equipment are controlled by a minicomputer which also suitably evaluates and presents the results. The different modules in our system can be connected in a number of different ways but here I would emphasize:

1) Hydraulic Scanning System
2) Rotating Sound Field Measuring System, and
3) Automatic Tube Testing System

with some examples from the use we have made of the systems.

Patent på automatisk kontrol af rør efter femten års kamp. (Automatic Inspection of Tube Dimension Patented After Fifteen Years of Fighting).

H.E. Gundtoft, Ingeniøren 12 no. 42 (1986) 50.

Risø patent fører til licenskontrakt med Japan og Vesttyskland. (Risø Patent Leads to Royalties from Japan and Western Germany).

H.E. Gundtoft, Risønyt no. 4 (1986) 3.

Additive Strengthening Mechanisms in Dispersion Hardened Polycrystals.

N. Hansen and B. Ralph, Acta Metall. 34 (1986) 1955-1962.

Tensile data from polycrystalline samples of copper dispersion strengthened by alumina are analysed. The basis of this analysis is to look at the strain range from 0.05 to 0.20 where the stress-strain curves show a parabolic hardening behaviour and are parallel to one another. The means by which the addition of strength components from various elements of the microstructure might explain this behaviour are investigated. It is shown that a linear combination of a matrix friction stress, and Orowan bowing stress, a matrix mean stress from the particles and a combined dislocation interaction term can explain this data and also the data from some aluminium-alumina materials. The dislocation interaction term, which dominates, is comprised of terms which cover the pure matrix work hardening, the hardening due to particles and due to the grain boundaries. This term is derived by summing the dislocation density contributions from each of these three sources. The type of additivity suggested here not only gives very good agreement with the stress-strain data but it also uses and is in accord with the experimental measurements of dislocation densities made using transmission electron microscopy.

Annealing Processes - Recovery, Recrystallization and Grain Growth.

N. Hansen, D. Juul Jensen, T. Leffers and B. Ralph (editors). Proceedings of the 7th Risø International Symposium on Metallurgy and Materials Science, Risø, 8-12 September 1986 (Risø National Laboratory, Roskilde, 1986). (601 pp.).

Deformation and Recrystallization Textures in Commercially Pure Aluminium.

N. Hansen and D. Juul Jensen, Met. Trans. A 17A (1986) 253-259.

The deformation and recrystallization textures of commercially pure aluminium (99.6%) containing large intermetallic particles ($FeAl_3$) are measured by neutron diffraction, and the orientation distribution functions (ODF's) are calculated. Sample parameters are the initial grain size (50 and 350 μm) and the degree of deformation (15 to 95% reduction in thickness by cold-rolling). The textural results are compared with microstructural observations and good correlations are found. The intermetallic particles may act as nucleation sites giving nuclei with a wide spread of orientations. Thereby the particles can have a randomizing effect on the textural development during recrystallis-

ation. In specimens deformed at medium degrees of deformation the randomizing effect of particles is maximum. At lower and higher degrees of deformation the effect of particles is less as other nucleation sites become more effective. In general, the randomizing effect of particles is limited due to a low growth rate of nuclei of random orientation compared with nuclei of other orientations.

Deformed and Recovered Microstructures in Pure Aluminium.
B. Bay and N. Hansen, In: Annealing Processes - Recovery, Recrystallization and Grain Growth. Proceedings of the 7th Risø International Symposium on Metallurgy and Materials Science, Risø, 8-12 September 1986. Edited by N. Hansen et al. (Risø National Laboratory, Roskilde, 1986) 215-220.

The microstructure of pure aluminium (99.998%) with a grain size of 130 μm was studied by transmission electron microscopy (TEM) after 30% cold rolling and after annealing in the temperature range 170°-300°C. A number of structural features were characterized in the cold worked state and their change during annealing was studied. Furthermore the growth of the subgrains was measured and coalescence events were observed to be part of the growth process. Finally, subgrain growth leading to the formation of recrystallization nuclei is discussed with regard to nucleation at the original grain boundaries and in the grain interiors, respectively.

Effect of Small Particles on Deformation and Recrystallization Textures of Aluminium.
N. Hansen and D. Juul Jensen, In: Annealing Processes - Recovery, Recrystallization and Grain Growth. Proceedings of the 7th Risø International Symposium on Metallurgy and Materials Science, Risø, 8-12 September 1986. Edited by N. Hansen et al. (Risø National Laboratory, Roskilde, 1986) 337-342.

The textural development has been investigated after cold-drawing (50 and 90%) and after recrystallization of aluminium-aluminium oxide specimens containing from $0.16 \cdot 10^{-2}$ to 0.4 vol.% aluminium oxide particles of a diameter less than 250 nm. The particle spacing varies within an order of magnitude and the textural changes are analyzed as a function of this parameter. A relatively large effect of particles on the concentration of the <111> component is observed both after cold-drawing and after recrystallization. These results are discussed on basis of the effect of particles on the microstructures in the deformed state and on the formation of recrystallization nuclei.

Grain Growth in Crystalline Materials.

R. Randle, B. Ralph and N. Hansen, In: Annealing Processes - Recovery, Recrystallization and Grain Growth. Proceedings of the 7th Risø International Symposium on Metallurgy and Materials Science, Risø, 8-12 September 1986. Edited by N. Hansen et al. (Risø National Laboratory, Roskilde, 1986) 123-142.

A review is presented of the processes and mechanisms of grain growth in materials. The presentation looks briefly at those factors which influence the course of grain growth but concentrates on the pinning effects of particles. It is shown that the interactions between migrating grain boundaries and incoherent particles may be adequately described in most cases by the model due to Zener and derivations thereof. For particles which are initially coherent with the matrix, the pinning is found to be much stronger and this effect is found to inhibit growth.

Grain Growth in Single-Phase and Particle-Containing Materials.

N. Hansen, S.E. Soliman and D. Juul Jensen, In: Proceedings of the Denmark-Israel Binational Symposium on High Temperature Materials, Jerusalem, 14-15 April 1986. Edited by A. Kaufman (Ministry of Science and Development, National Council for Research and Development, Jerusalem 1986). 13 pp.

A review is presented of the processes and mechanisms of grain growth in crystalline materials. Many factors influence the grain growth and the paper considers briefly the effect of porosities, texture, solute elements, and dispersed particles. Theories for grain growth are discussed and related to experimental findings in single-phase and particle-containing materials.

Low Energy Dislocation Structures due to Unidirectional Deformation at Low Temperatures.

N. Hansen and D. Kuhlmann-Wilsdorf, Mater. Sci. Eng. 81 (1986) 141-161.

The line energy of dislocations is $\{Gb^2f(\nu)/4\pi\}\ \ln(R/b)$ with R the range of the dislocation stress field from the axis. This equation implies that quasi-uniform distributions are unstable relative to dislocation clusters in which neighboring dislocations mutually screen their stress fields, correspondingly leaving the major fraction of the volume free of dislocations. The value of R decreases in the following order: pile-ups to dipolar mats, Taylor lattices,

tilt and dipolar walls to dislocation cell structures. This is the same order in which dislocation structures tend to develop with increasing dislocation density and hence increased dislocation interactions, leading to the corresponding energy decrease per unit length of dislocation line. Taking into consideration also the longer-range "termination stresses" of finite dislocation boundaries, and minimizing the total energy, explains the size dependence of cells on stress as well as the occasionally observed pattern of rectangular cells with alternating left-right rotation about a common axis. Energy minimization further explains the transition from stage I to II in f.c.c. metals. The following topics which are at the present frontier of research are considered in this paper: (i) the sum of the energy stored in the dislocation line energy and the longer-range stresses is significantly smaller than some recently reported experimental values; (ii) subdivision of cells is discussed on the basis of observations in rolled aluminum showing a "hierarchical" cell structure; (iii) suggestions are made to account for microband formation on the basis of energy minimization. Finally, the relationship between surface markings and the underlying dislocation structure is discussed and related to slip processes taking place during uniaxial deformation.

Role of Dislocation Walls and Grain Boundaries in Void Formation During Early Stages of Fast Neutron Radiation.

A. Horsewell and B. Singh, In: Effects of Radiation on Materials: Twelfth International Symposium, ASTM, STP 870. Edited by F.A. Garner and J.S. Perrin. (American Society for Testing and Materials, Philadelphia, 1986) 248-261.

High purity aluminium was used to study the details of microstructural evolution during early stages of neutron irradiation. Aluminium specimens were irradiated at 120°C to fluences between $2 \cdot 10^{21}$ and $1 \cdot 10^{24}$ n/m^2(E>0.1MeV). Transmission electron microscopy (TEM) investigations demonstrated that, even in fully annealed material, irradiation-induced dislocations and voids evolve heterogeneously. In addition, voids and dislocations were found to segregate such that the groups of voids and dislocations are spatially separated from each other. This kind of heterogeneity and segregation is further enhanced by the introduction of microstructural heterogeneity (in the form of dislocation walls) prior to irradiation.

Another form of heterogeneity was found to occur in a relatively wide band in the vicinity of the void denuded zone along grain boundaries; in this region, both formation and growth of voids were enhanced compared to that observed in the grain interior.

It is argued that these results cannot be rationalized in terms of a conventional biasdriven mechanism operating in a continuous sink medium. Both cell size and grain boundary effects would indicate an unusually high rate of transport of self-interstitial atoms from cell- and grain-interiors to cell walls and grain boundaries.

A Determination of the Texture of a Directionally Solidified Sample of High-Purity Copper.

E. Grant, D. Juul Jensen and B. Ralph, J. Mater. Sci. 21 (1986) 1688-1692.

A study making a combined use of neutron diffraction and selected-area electron channelling to determine the solidification texture in a high-purity copper sample is described. Good correlation between the techniques is shown with both demonstrating a strong [100] fibre texture in the directionally solidified rod.

A Kinetic Model for Recrystallization of Commercially Pure Aluminium.

D. Juul Jensen and N. Hansen, In: Annealing Processes - Recovery, Recrystallization and Grain Growth. Proceedings of the 7th Risø International Symposium on Metallurgy and Materials Science, Risø, 8-12 September 1986. Edited by N. Hansen et al. (Risø National Laboratory, Roskilde, 1986) 379-384.

Grains of different orientations may nucleate and grow differently. This has been incorporated in a computer model for recrystallization. The model has been used to simulate the recrystallization behaviour of heavily deformed commercially pure aluminium. The differences between the model calculations and the experimental results are discussed with reference to the model assumptions.

Fast Texture Measurement by Neutron Diffraction Using a Linear Position Sensitive Detector.

D. Juul Jensen, In: Experimental Techniques of Texture Analysis. Edited by H.J. Bunge. (DGM Informationsgesellschaft Verlag, Oberursel, 1986) 217-228.

A technique for fast in-situ texture determination by neutron diffraction is described. The application of the technique is illustrated by in-situ kinetic investigations of recrystallization and grain growth in various polycrystalline metals. Its potential for problems related to metallurgical practice is demonstrated by an approach of texture tailoring in commercially pure aluminium.

Hurtig teksturmåling med neutronspredning (Fast Texture Measurement by Neutron Scattering).

D. Juul Jensen, In: Måling af materialeegenskaber (Measurement of Materials Properties). Dansk Metallurgisk Selskabs Vintermøde, Risø, 6-8 January 1986. Edited by H. Lilholt and G. Skjeldsager. (Dansk Metallurgisk Selskab, Lyngby, 1986) 135-154.

A technique for a fast in-situ texture determination by neutron diffraction is described. By an on-line recording of texture changes, information about metallurgical processes can be obtained if such processes are accompanied by a change in texture. This is illustrated by a kinetic investigation of recrystallization of commercially pure aluminium. Finally, the potential of the technique for problems related to metallurgical practice is demonstrated by an approach of texture tailoring.

Neutron Diffraction Methods for the Measurement of Texture Development During Grain Growth.

E. Grant, D. Juul Jensen, N. Hansen, B. Ralph and W.M. Stobbs, In: Annealing Processes - Recovery, Recrystallization and Grain Growth. Proceedings of the 7th Risø International Symposium on Metallurgy and Materials Science, Risø, 8-12 September 1986. Edited by N. Hansen et al. (Risø National Laboratory, Roskilde, 1986) 329-336.

The techniques for texture determination using neutron diffraction developed at Risø have been applied in a study of the change in texture associated with grain growth in 99.999 wt.% pure copper rods. While these techniques would appear to allow a quasi-dynamic study to be made relatively easily extinction losses can be a problem, particularly when grain growth is not normal as is the case for the system examined. In the present study extinction corrections were made by a normalisation procedure where dynamic curves measured for a stable texture component were used as reference. The corrected results for the copper examined indicate that the <111> fibre texture present after recrystallisation strengthens during grain growth, with an activation energy for the rate controlling process of 110 kJ mol^{-1}.

Structure and Texture Evolution During the Recrystallization of Particle Containing Materials.

F.J. Humphreys and D. Juul Jensen, In: Annealing Processes - Recovery, Recrystallization and Grain Growth. Proceedings of the 7th Risø International Symposium on Metallurgy and Materials Science, Risø, 8-12 September 1986. Edited by N. Hansen et al. (Risø National Laboratory, Roskilde, 1986) 93-106.

The effect of second-phase particles on the deformation structure and on the basic recrystallisation processes of nucleation and growth is reviewed, and models which predict the recrystallised grain size are discussed. The recrystallisation texture of the alloys containing large (>1μm) particles is shown to depend on a balance between the relatively randomly oriented grains nucleated near particles, and grains nucleated in the matrix. A fine particle dispersion generally results in a recrystallisation texture which is close to that of the deformed material.

Calculations for Piezoelectric Ultrasonic Transducers.

H. Jensen, Risø-R-536 (Risø National Laboratory, Roskilde, 1986) 97 pp.

Analysis of piezoelectric ultrasonic transducers implies a solution of a boundary value problem, for a body which consists of different materials, including a piezoelectric part. The problem is dynamic at frequencies, where a typical wavelength is somewhat less than the size of the body. Radiation losses as well as internal losses may be important. Due to the complexity of the problem, a closed form solution is the exception rather than the rule. For this reason, it is necessary to use approximate methods for the analysis.

Equivalent circuits, the Rayleigh-Ritz method, Mindlin plate theory and in particular the finite element method are considered. The finite element method is utilized for analysis of axisymmetric transducers. An explicit, fully piezoelectric, triangular ring element, with linear variations in displacement and electric potential is given. The influence of a fluid half-space is also given, in the form of a complex stiffness matrix.

A special stacking procedure, for analysis of the backing has been developed. This procedure gives a saving, which is similar to that of fast Fourier transform algorithm, and is also well-suited for analysis of finite and infinite waveguides. Results obtained by the finite element method are shown and compared with measurements and exact solutions. Good agreement is obtained. It is concluded that the finite element method can be a valuable tool in analysis and design of ultrasonic transducers.

Fission Product Behavior in High-Burnup Water Reactor Fuel Subjected to Slow Power Increases.

P. Knudsen, C. Bagger, H. Carlsen, I. Misfeldt and M. Mogensen, Nucl. Technol. 72 (1986) 258-267.

Data are presented on fission gas release for UO_2-Zr fuel pins that were subjected to slow power increases late in life. These tests were performed with fuel pins that had been previously irradiated to average burnups of 27000 to 35000 MWd/ton U (peak pellet 43700 MWd/ton U). The subsequent power increases were from 301 to 444 W/cm (peak pellet), and the hold time was 24 h, with one test at 72 h.

Emphasis was given to extensive axially and radially local measurements, rather than to integral pin data. Cross-sectional releases increased with transient powers above 350 W/cm; at 415 W/cm they seemed to saturate at 40 to 45% within 24 h. Radially local releases started at calculated local temperatures at ~ 700°C, reaching a constant level of 90-95% above 1100°C. Local swelling appeared to begin at ~ 650 to 700°C, with maximum swelling levels at 10%. From the observed transient release data, an effective diffusion coefficient could be calculated that was more than three orders of magnitude higher than commonly accepted values.

The Risø Fission Gas Projects.

P. Knudsen, In: Performance of Fuel and Cladding Material Under Reactor Operating Conditions. Proceedings of a conference in Karlsruhe, 28-29 November 1985. Edited by G. Mühling and W. Dietz. (Kernforschungszentrum Karlsruhe, Karlsruhe, 1986) 21-29.

The desire to improve fuel utilization has created a growing interest in extending the burnup of LWR fuel. As a result, fuel performance data are needed at burnup levels well in excess of 30,000 MWd/tU. Fission gas release is one of the important factors, especially for power increases (transients) late in life, because even moderate increases can then lead to important releases.

The combination of a number of capabilities related to fuel performance testing enables Risø to execute major projects to study the various aspects of fission gas release in UO_2 fuel at high burnup. These capabilities include: the fuel testing facilities at DR3; urique instrumentations for fuel pins; refabrication and other specialized hotcell techniques; experience in fuel performance evaluation and project management. The paper presents an overview of the two Risø fission gas projects carried out 1980-1986 and a brief outline of the new program planned for 1986-90.

The following are some conclusions from the work performed so far:

(a) Fission gas release and swelling in power transients are very high for high-burnup.
(b) The effective diffusion coefficient for fission gas appears to be much higher in power transients than in steady-state conditions.
(c) Instrumentation of high-burnup test fuel with pressure transducers gives very detailed information on the fission gas release during transient tests.
(d) Extensive hotcell examinations with various types of local measurements provide valuable input to benchmarking and further development of fission gas release models.

Effects of Heterogeneous Sink Distribution on Void Swelling.

T. Leffers, B.N. Singh, A.V. Volobuyev and V.V. Gann, Phil. Mag. A, 53 (1986) 243-257.

Swelling rates are calculated for two types of material with heterogeneous distributions of dislocations and voids, namely copper irradiated with neutrons to low dose at 250°C and heavily cold-worked copper irradiated with 1 MeV electrons in a HVEM at 250°C. Both materials are considered to consist of non-interacting spherical components with a wall and an inner cell with different dislocation and/or void densities. We subdivide the sphere (wall plus cell) in a number of concentric shells and find a quasi-static solution for the interstitial and vacancy concentrations in the different shells by a finite-difference method. From these concentrations the local and the average swelling rates are calculated. The effect of the heterogeneities in sink distribution on swelling rate and the dependence of this effect on various structural parameters are investigated. We find that a heterogeneous sink distribution may increase or decrease the swelling rate relative to the swelling rate for a homogeneous distribution of the sinks depending on the structural parameters and the irradiation conditions. For neutron-irradiated copper neither the calculated swelling rate nor its spatial variation agree with the experimental observations. For cold-worked copper, on the other hand, there is reasonable agreement between calculations and experiments.

Evaluation of the Effect of Initial Texture on the Development of Deformation Texture.

T. Leffers and D. Juul Jensen, Textures Microstruct. 6 (1986) 231-254.

We describe a computer procedure which allows us to introduce experimental initial textures as starting conditions for texture simulation (instead of a theoretical random texture). We apply the procedure on two batches of copper with weak initial textures and on fine-grained and coarse-grained aluminium with moderately strong initial textures. In copper the initial texture turns

out to be too weak to have any significant effect. In aluminium the initial texture has a very significant effect on the simulated textures - similar to the effect it has on the experimental textures. However, there are differences between the simulated and the experimental aluminium textures that can only be explained as a grain-size effect. Possible future applications of the procedure are discussed.

Influence of Inhomogeneous Sink Distribution on Vacancy Swelling.
A.V. Volobuev, V.V. Gann, T. Leffers and B.N. Singh, Voprosy Atomnoj Nauki i Tekhniki, Series Fizika Radiatsionykh Povrezhdenii i Radiatsionnoye Materialovedenie, 38 No. 1 (1986) 49-58.

Modelling of Recrystallization Kinetics on Stereological Basis.
T. Leffers, In: Annealing Processes - Recovery, Recrystallization and Grain Growth. Proceedings of the 7th Risø International Symposium on Metallurgy and Materials Science, Risø, 8-12 September 1986. (Risø National Laboratory, Roskilde, 1986) 427-436.

Recrystallization kinetics are investigated with a geometrical computer model which, within the given set of assumptions, produces a stereologically correct simulation of the recrystallization process. In the present work the model provides the β values in the Avrami-type equation for a number of simple nucleation and growth modes, with the aim of establishing the basic conditions for the low β values found in the great majority of experimental investigations.

Måling af indre spændinger med neutrondiffraktion (Measurement of Internal Stresses with Neutron Diffraction).
T. Leffers and D. Juul Jensen, In: Måling af materialeegenskaber (Measurement of Materials Properties). Dansk Metallurgisk Selskabs Vintermøde, Risø, 6-8 January 1986. Edited by H. Lilholt and G. Skjelsager. (Dansk Metallurgisk Selskab, Lyngby, 1986) 239-247.

The general background for measurement of internal stresses with diffraction methods is outlined, and the advantages of neutron diffraction as compared with X-ray diffraction in stress measurements are pointed out (penetration into the bulk, variable wave length). The difference between microscopic and

macroscopic internal stresses and the corresponding differences in the requirements to be met by the measuring equipment for the two cases are described. Two new concepts for equipment for measurement of the two types of internal stresses are presented.

Fibre Reinforced Materials for High Temperature Applications.
H. Lilholt, In: High Temperature Materials. Proceedings of the Denmark-Israel Binational Symposium on High Temperature Materials, Jerusalem 13-18 April 1986. Edited by A. Kaufman (Ministry of Science and Development, National Council for Research and Development, Jerusalem, 1986) 16 pp.

Creep of fibre reinforced materials is classified into three groups, which depend on the fibre orientation and the related internal stress state of the material. The models for these groups are presented, and general trends in creep strength are indicated.

Glasfiberforstærket polyester til vindmøllevinger (Glass Fibre-Reinforced Polyester for Wind Turbine Blades).
H. Lilholt, In: Vinger og Materialer (Wingblades and Materials). Proceedings of the conference Vinger og materialer, Risø National Laboratory, 20 March 1986. (Dansk Selskab for Materialprøvning og -forskning, 1986). Materialnyt No. 2 (1986) 15-30.

The properties of relevance to the design of wingblades for windturbines are presented: density, stiffness, vibration frequency, bending properties, fatigue strength and design rules.

Glasfiberforstærket polyester - et konstruktionsmateriale til Flådens FLEX 300 skibe (Glass Fibre-Reinforced Polyester - A Construction Material for the Navy's FLEX 300 Ships).
H. Lilholt, Tidsskr. Søvæsen, 157 (1986) 163-206.

The relevant properties of glass fibre reinforced polyester are reviewed: basic properties of glass fibre, properties of polyester (and other polymers); density of the composite material; its stiffness, strength, fracture toughness, fatigue strength and behaviour in water environment.

Long Term Characterization of Composite Materials.

H. Lilholt, In: Advanced Materials Research and Development for Transport-Composites. Proceedings of the Conference on Advanced Materials Research and Development for Transport-Composites, Strasbourg, 26-29 November 1985. Edited by P. Lamicq et al. (Les Editions de Physique, Les Ulis Cedex, France, 1986) 259-273.

Models for the creep behaviour of fibrous composite materials are reviewed. The models describe the creep rate and the strength contributions during creep. In this presentation an attempt is made to classify the composites into three groups: aligned composites, off-axis composites and general composites. The models include cases where the fibres remain rigid in a creeping matrix and cases where both fibres and matrix are creeping, and also the transition between these two (extreme) cases. An attempt is made to include multiaxial creep in the models; this becomes particularly relevant for off-axis and general composites.

Måling af materialeegenskaber (Measurement of Materials Properties).

H. Lilholt and G. Skjelsager (editors). Proceedings of Dansk Metallurgisk Selskabs Vintermøde, Risø National Laboratory, 6-8 January 1986. (Dansk Metallurgisk Selskab, Lyngby, 1986). 409 pp.

The book contains the lectures given at the Wintermeeting of the Danish Metallurgical Society, on the subject: measurement of materials properties.

Måling af stivhed og styrke af anisotrope materialer (Measurement of Stiffness and Strength of Anisotropic Materials).

H. Lilholt, In: Måling af materialeegenskaber (Measurement of Materials Properties). Dansk Metallurgisk Selskabs Vintermøde, Risø National Laboratory, 6-8 January 1986). Edited by H. Lilholt and G. Skjelsager. (Dansk Metallurgisk Selskab, Lyngby, 1986) 249-269.

The stiffness and strength of fibre reinforced materials are measured by three point bending; the span is varied to obtain various combinations of tensile and shear deformation.

Tensile and Shear Properties of Fibre-Reinforced Materials.

H. Lilholt, In: Mechanical Characterization of Fibre Composite Materials. Proceedings of the Conference on Mechanical Characterization of Fibre Composite Materials, Aalborg University, 3-4 June 1986. Edited by R. Pyrz. (Aalborg Universitet, Aalborg, 1986) 129-146.

The tensile and shear properties are derived from three point bending tests, where the span is varied to give various combinations of tensile and shear deformations. Both stiffness and strength of fibre composites are measured by this method.

Vinger og materialer (Wingblades and Materials).

H. Lilholt (editor). Proceedings of the conference Vinger og materialer, Risø National Laboratory, 20 March 1986 (Dansk Selskab for Materialprøvning og -forskning, København, 1986) Materialnyt No. 2 (1986) 88 pp.

The book contains the 8 presentations given on wingblades for windturbines and the materials problems related to the design of wingblades.

Plastbaserede kompositmaterialer med lange fibre (Advanced Fiber Reinforced Plastics).

Aa. Lystrup, Dansk Tekn. Tidsskr. 110 No. 10 (1986) 10-16.

The properties of advanced fibre reinforced plastic and its various applications are discussed, and the principles in design and building of laminates are described. Autoclave curing and filament winding, as the most common processes for fabrication of advanced fibre reinforced plastics, are also discussed. Finally, the state of the art within the Danish industry is reviewed.

Determination of Fission Products in Irradiated Fuel by X-Ray Fluorescence.

M. Mogensen, J. Als-Nielsen and N.H. Andersen, Risø-M-2599 (Risø National Laboratory, Roskilde, 1986) 21 pp.

X-ray fluorescence is a well established analytical tool for measuring element composition of fairly large (~ 5 cm^2) "cold" samples. A version of this technique has been developed for analysis of radial distribution of fission pro-

ducts Xe, Cs and Ba in irradiated UO_2 fuel samples. About 0.1 mm thin slices of fuel pellets (full cross sections) are irradiated by 50 keV X-rays. The intensity of the Xe, (Cs, Ba) $K\alpha$ fluorescence radiation generated is measured by means of a Ge detector fitted with a collimator. The slit is 0.5 mm wide in the scanning direction and 2 m. long. The measured Xe $K\alpha$ X-ray intensities are converted to absolute concentrations by comparison with the intensity from a Xe gas standard. In the case of Cs and Ba solid standards may be used. The X-ray flourescence analysis is compared with other techniques used to obtain radial fission product profiles. It is shown how a combination of X-ray fluorescence and electron probe micro analysis is able to reveal the amount of Xe in the grain boundary porosities.

Frequency Response Analysis: A Tool for the Study of Metal Degradation.

M. Mogensen, Proceedings of the 10th Scandinavian Corrosion Congress, Stockholm, 2-4 June 1986. (Swedish Corrosion Institute, Stockholm, 1986) 255-261.

Many theoretical treatments of ac frequency response analysis on corroding electrodes have been published during recent years. Usually it is assumed that an electrode behaves like a resistor coupled in parallel to a capacitor. When the impedance of such a system is plotted in a Nyquist diagram a semicircle is obtained. However, actual measurements very seldom turn out to be a semicircle. Some very different examples on what the results may look like are given and qualitative explanations outlined. The examples are: 1) a lithium electrode in a thionyl chloride solution, 2) a synthetic $FeCr_{25}Al_5$ alloy with an aluminium oxide layer at room temperature and at 810°C, 3) a plain carbon steel in a 3.5% NaCl aqueous solution, and 4) the same steel covered with a 100µm layer of alkyd synthetic resin in the same solution. Examples 3) and 4) were acquired by means of a probe allowing the impedance to be measured locally on a large surface. The automatic data acquisition system for ac impedance as well as dc electrochemical measurements used in the Risø Metallurgy Department is briefly described.

Måling af nogle vandige opløsningers permittivitet (Measurement of Permittivity of Some Aqueous Solutions).

M. Mogensen, Report to Novo Industri A/S, (Risø National Laboratory, Roskilde, 1986). Available on request (13 pp.).

The permittivity (dielectric constant) of aqueous solutions with a specific conductivity of ~ $3 \cdot 10^{-4}$ S/cm were measured. A solution was placed in a parallel plate "capacitor" (or cell) of titanium, and the impedance of the cell was measured for several distances between the plates. By this procedure and by a very careful calibration of the impedance analyser it was possible to determine the permittivity of such solutions with an accuracy of about 1% even though the phase shifts were in the order of 1°, only.

Tynde fastelektrolytlag på lithiumelektroder (Thin Solid Electrolyte Layers in Lithium Electrodes).

M. Mogensen and F.W. Poulsen, In: Generel og anvendt elektrokemi i Danmark (General and Applied Electrochimistry in Denmark). Extended abstracts of the symposium Generel og anvendt elektrokemi i Danmark, Danmarks Tekniske Højskole, 9 april 1986. (Dansk Elektrokemisk Forening, Selskabet for Analytisk Kemi, Lyngby, 1986) 26-27.

The main results of characterizing the thin solid electrolyte layers on Li electrodes in $SOCl_2$ and in SO_3 solutions were presented and the related subjects to be studied in the near future were explained.

Metallurgy Department Progress Report for the Period 1 January to 31 December 1985.

A. Schrøder Pedersen and J.B. Bilde-Sørensen (editors). Risø-R-537 (Risø National Laboratory, Roskilde, 1986) 93 pp.

The activities of the Metallurgy Department at Risø during 1985 are described. The work is presented in four chapters: General Materials Research, Technology and Materials Development, Chemical and Electrochemical Energy Research and Development, and Fuel Elements. A survey is given of the Department's participation in international collaboration and of its activities within education and training. A list (with abstracts) of publications and lectures by the staff during 1985 is included.

The Effect of Cycling in Impure Hydrogen on the Hydrogen Storage Capacity of Magnesium Powder.

A. Schrøder Pedersen, B. Vigeholm, J. Kjøller and B. Larsen, In: Hydrogen Energy Progress VI. Proceedings of the 6th World Hydrogen Energy Conference, Vienna, 20-24 July 1986. Edited by T.N. Veziroglu et al. (Pergamon Press, Oxford, 1986) 935-942.

In a series of continuous cycling experiments magnesium powder with average particle diameter around 65 μm was exposed to hydrogen gases containing oxygen and nitrogen. The absorption and the desorption were measured over approx. 17 minutes each at a temperature of 375°C with small variations caused by the heat of reaction. The hydrogen gases used were N57 (99.9997%), H_2 + approx. 0.5% O_2 and H_2 + approx. 0.5% N_2. In pure hydrogen approx. 85% of the magnesium reacted within the absorption period to form MgH_2. When 0.5% O_2 or

N_2 was added a prompt drop took place. When the Mg powder was exposed to 0.5% N_2 from the start, the uptake of hydrogen gradually rose to 35% over 50 cycles. The sample exposed to 0.5% O_2 suffered a permanent loss of approximately 20% of the initial capacity. The effect of 0.5% N_2 was similar but less pronounced. The temporary and permanent effects of the impurities are discussed.

Undersøgelse af litteratur om lagring af naturgas ved adsorption (A Study of the Available Literature on Adsorptive Storage of Natural Gas).

A. Schrøder Pedersen, Risø-M-2590 (Risø National Laboratory, Roskilde, 1986) 30 pp. Also published in report to the Danish Ministry of Energy, EFP 85-1243/85-8 (Energy Research Laboratory, Odense, 1986) 45 pp.

This report was prepared as Risø's contribution to a joint LFE/Risø reporting on the EFP-86 project "Utilization of Zeolithes and Other Adsorbents for Storage of Natural Gas". The report resulted from an investigation of the published literature relevant to the subject. The publications are briefly reviewed and the most important results in the respective works are given. The report gives complete references to all reviewed literature. The best result found with regard to the density of stored gas was 3.9 mmol/cm^3 adsorbent at 3.6 MPa and 296°C, corresponding to 99 mg/g for this material, a carbon black.

Composite Li-Conducting Solid Electrolytes.

F.W. Poulsen, Risø-M-2540 (Risø National Laboratory, Roskilde, 1986) 94 pp.

This is the final report for a joint project between the Metallurgy Department and Physics Department at Risø National Laboratory. Results on alumina based composite electrolytes containing LiI, LiBr and Li_3N are summarized. Experimental methods covered are ac-impedance, X-ray and neutron diffraction, SANS, BET-measurements and electron microscopy. The influence on the observed ionic conductivity from different fabrication methods, source of chemicals, specific surface area of alumina, composition, and temperature has been studied. Reprints of 6 published papers are included in the present report.

Prospektering af faststof Li-ionledere (Searching for Li-Conducting Solid Electrolytes).

F.W. Poulsen, In: Generel og anvendt elektrokemi i Danmark (General and Applied Electrochemistry in Denmark). Extended abstracts of the symposium Generel og anvendt elektrokemi i Danmark, Danmarks Tekniske Højskole, 9 April 1986. (Dansk Elektrokemisk Forening, Selskabet for Analytisk Kemi, Lyngby, 1986) 14-15.

A prerequisite for making rechargeable Li-batteries is a good conducting and stable Li electrolyte. The present paper outlines different research strategies in the field of solid electrolytes.

Raman Spectrum of the Solid Electrolytes $LiI \cdot H_2O$ and $LiI \cdot D_2O$.

F.W. Poulsen, J. Raman Spectrosc. 17 (1986) 189-191.

The Raman spectra of cubic $LiI \cdot H_2O$ and $LiI \cdot D_2O$ have been revised. The spectra reveal only internal and librational modes of $H_2O(D_2O)$. The isotopic ratios, ν_H/ν_D, are in the range 1.33-1.78.

Dislocation and Void Segregation in Copper During Neutron Irradiation.

B.N. Singh, T. Leffers and A. Horsewell, Phil. Mag. A 53 (1986) 233-242.

High-purity (99.99%) and fully annealed copper specimens have been irradiated in the DR-3 reactor at Risø to doses of 1×10^{22} and 5×10^{22} neutrons (fast) m^{-2}(2×10^{-3}dpa and 1×10^{-2}dpa, respectively); the irradiation experiments were carried out at 250°C. The irradiated specimens were examined by transmission electron microscopy. At both doses, the irradiation-induced structure was found to be highly segregated; the dislocation loops and segments were present in the form of irregular walls and the voids were distributed between these walls. The dislocation walls were practically free of voids and generally had a void-denuded zone along them. The density of dislocations (loops and segments) was very low in the region containing voids (i.e. between the dislocation walls). Even with this low dislocation density, the void swelling rate was very high (~ 2.5% per dpa). The implications of the segregated distribution of sinks for void formation and growth are briefly discussed. It is pointed out that the present observations cannot be understood in terms of the conventional bias-driven swelling mechanism.

Effects of 600 MeV Proton Irradiation on Nucleation and Growth of Precipitates and Helium Bubbles in a High-Purity Al-Mg-Si-Alloy.

B.N. Singh, T. Leffers, M. Victoria, W.V. Green and D. Gavillet, J. Nucl. Mater. 141-143 (1986) 743-747.

Solution treated specimens of a high-purity Al-0.75% Mg-0.42% Si alloy were irradiated with 600 MeV protons at 150 and 240°C to a dose level of 0.47 and 0.55 dpa, respectively. Mg_2Si-type precipitates formed during irradiation at 150 and 240°C; at 240°C, however, a large number of precipitates seem to have dissolved during the later stages of irradiation. Thermally aged reference specimens have also been investigated. The needle-shaped precipitates in the aged and the irradiated specimens lie along the <100> matrix directions. At 150°C bubbles were observed only at grain boundaries whereas at 240°C bubbles were seen in the grain interior as well as at the grain boundaries. Long rows of bubbles were observed with the same orientation in the matrix as the precipitate needles. Grain boundary bubbles were found to grow faster in the Al-Mg-Si alloy than in high-purity aluminium.

Gas Accumulation at Grain Boundaries During 800 MeV Proton Irradiation of Aluminium and Aluminium Alloys.

B.N. Singh, A. Horsewell, W.P. Sommer and W. Lohmann, J. Nucl. Mater. 141-143 (1986) 718-722.

Samples of pure aluminium (99.9999%) and commercial Al-2.7% Mg (AlMg3) and Al-1.1% Mg-0.5% Si (A16061) alloys were irradiated with 800 MeV protons at the Los Alamos Meson Physics Facility (LAMPF) at a temperature between 40 and 100°C to a maximum dose of 0.2 dpa. Transmission electron microscopy (TEM) showed a complete absence of voids or bubbles in the grain interiors of the aluminium and the aluminium alloys. Bubbles were clearly visible by TEM at grain boundaries in pure Al and the AlMg3 alloy; but bubbles were not visible in the A16061 alloy. The bubble density in the AlMg3 alloy was considerably higher than in pure Al. The amount of gas accumulation at grain boundaries was found to depend on gas generation rate, alloying and cold-work microstructure.

Gas Diffusion and Temperature Dependence of Bubble Nucleation During Irradiation.

A.J.E. Foreman and B.N. Singh, J. Nucl. Mater. 141-143 (1986) 672-676.

The continuous production of gases at relatively high rates under fusion irradiation conditions may enhance the nucleation of cavities. This can cause dimensional changes and could induce embrittlement arising from gas accumulation on grain boundaries. Computer calculations have been made of the diatomic nucleation of helium bubbles, assuming helium to diffuse substitutionally, with radiation-enhanced diffusion at lower temperatures. The calculated temperature dependence of the bubble density shows excellent agreement with that observed in 600 MeV proton irradiations, including a reduction in activation energy below $T_m/2$. The coalescence of diatomic nuclei due to Brownian motion markedly improves the agreement and also provides a well-defined terminal density. Bubble nucleation by this mechanism is sufficiently fast to inhibit any appreciable initial loss of gas to grain boundaries during the nucleation period, provided that incubation effects do not occur.

On Transport of Helium to Grain Boundaries During Irradiation.
B.N. Singh and A.J.E. Foreman, Risø-M-2612 (Risø National Laboratory, Roskilde, 1986) 24 pp. To appear in ASTM Special Technical Publications.

The rate of accumulation of helium at grain boundaries is one of the important parameters determining the integrity and lifetime of the structural components of a fusion reactor. A diffusion calculation is made of the flux of helium to a grain boundary. The flux is found to depend on the gas production rate, the width of the cavity denuded zone and the cavity sink strength in the grain interior. The calculated accumulation of helium is in good agreement with the measured gas content of grain boundaries in Al, PE16 and 316 stainless steel. The flux of helium to grain boundaries increases with helium generation rate but the increase is "less than proportional" to the generation rate. The loss of helium to grain boundaries during the nucleation of the bubbles within the grains has been estimated; no great loss is expected to occur. However, the loss would be considerably enhanced if any delay in bubble nucleation were to occur due to incubation effects. The role of material variables is found to be difficult to predict at present.

Isothermal Grain-Growth Kinetics in UO_2.
S.E. Soliman, N. Hansen, I. Misfeldt, J.G. Rasmussen and O. Toft Sørensen, In: Annealing Processes - Recovery, Recrystallization and Grain Growth. Proceedings of the 7th Risø International Symposium on Metallurgy and Materials Science, Risø, 8-12 September 1986. Edited by N. Hansen et al. (Risø National Laboratory, Roskilde, 1986) 553-560.

The grain growth of UO_2 pellets isothermally annealed at temperatures ranging from 1600 to 1900°C was investigated. Three different fuel batches were used. The deviation in grain size and porosity from pellet to pellet and within the same pellet was studied. The average grain size, the pore size and the total volume percent porosity were measured after each annealing. The three-dimensional grain and pore size distributions were studied and a statistical analysis was made for an accurate determination of small changes in the grain size. The results have been interpreted by using the empirical grain-growth equation $D^n - D_o^n = K_o t \exp(-Q/RT)$. The exponent "n" was found to be 4 and the activation energy of the three fuel batches was found to be 450 kJ/mole. In general the grain growth can be described by using the formula:

$$D^4 - D_o^4 = 5.5 \times 10^{14}\ t \exp(-54119/T)$$

where t is the annealing time (h) and T is the absolute temperature.

Eine Methode zum Polieren von Probekanten bei Faserverbundwerkstoffproben (A Method for Edge Polishing of Fiber Composite Tensile Test Specimens).

H. Toftegaard, Structure (Struers Metallographische Neuheiten) No. 13 (1986) 14-15 (Struers A/S, København, 1986).

The abstract appeared in the annual report for 1985.

The Effect of Stacking Sequence and Width on the Properties of Carbon/Epoxy Angle-Ply.

H. Toftegaard, In: Advanced Materials Research and Development for Transport-Composites. Proceedings of the Conference on Advanced Materials Research and Development for Transport-Composites, Strasbourg, 26-29 November 1985. Edited by P. Lamick et al. (Les Editions de Physique, Les Ulis Cedex, France, 1986) 53-59.

The abstract appeared in the annual report for 1985.

Sikkerhedsvurdering af magnesiumhydrid i energilagring (Safety Evaluation of Magnesium Hydride in Energy Storage).

B. Vigeholm, Risø-M-2609 (Risø National Laboratory, Roskilde, 1986) 50 pp.

Investigations of the magnesium/magnesium hydride system and its potential in energy storage have been carried out at Risø National Laboratory for some years. The results of this research indicate that the system is technically feasible, at least in some applications. The lack of available information of the hazards involved in handling magnesium hydride powder might however be a serious barrier to implementation. In this work a series of experiments relevant to handling and storage of magnesium hydride is reported. With a comparable magnesium powder as reference 100 - 400 g hydride powder was ignited under various conditions and the temperature in and over the powder mass recorded. Also the probable reaction of powder with water was investigated. The general conclusion is that magnesium hydride reacts in a way, very similar to magnesium and should be handled accordingly, allowing though for the potential release of hydr[illegible] during non-combusting heating.

LECTURES

Fremstilling af keramiske tyndfilm ved sputtering (Fabrication of Thin Ceramic Films by Sputtering).

N.H. Andersen, presented to Dansk Selskab for Materialprøvning og -forskning, Danmarks Tekniske Højskole, 22 May 1986. (Not available).

Quasielastic Diffuse Neutron Scattering Studies of the Defect Structure in Oxygen Conducting Yttria-Stabilized Zirconia.

N.H. Andersen, K.N. Clausen, M. Hackett, W. Hayes, J.E. MacDonald, R. Osborn and M. Hutchings, presented to the Danish Physical Society, Spring Meeting, Nyborg Strand, May 1986. (Not available).

ZrO_2 is an oxygen conductor which in the high temperature α-phase above 2370°C has the fluorite type of fcc-structure. The fast ionic conducting α-phase may be stabilized down to room temperature on doping with e.g. CaO, Sc_2O_3 and Y_2O_3. These stabilized materials have already been used for practical purposes, e.g. in oxygen monitors. Quasielastic diffuse neutron scattering studies have been performed on a number of compositions of Y_2O_3-doped ZrO_2 with the purpose of determining the defect structure responsible for the ionic conduction mechanisms. The defect structure consists of two components. One consisting of small areas without oxygen vacancies but with distortions of the anion lattice similar to the tetragonal β-phase of ZrO_2. The other contains arrays of defect clusters aligned along the (112) direction. Each defect cluster consists of two oxygen vacancies separated by $\langle a/2, a/2, a/2 \rangle$ in direct space and surrounded by relaxed nearest neighbour ions. The dynamical properties of the defect structure have been studied and they are discussed in relation to their influence on the ionic conduction process.

Tyndfilmmaterialer til elektrokemiske anvendelser (Thin Film Materials for Electrochemical Applications).

N.H. Andersen, J.J. Bentzen, J.B. Bilde-Sørensen, O. Eg, J.K. Kjems, L. Nordentoft, F.W. Poulsen and T.R. Strauss, presented at a FTU-Seminar, Danmarks Tekniske Højskole, August 1986. Available on request. (6 pp).

Statisk afprøvning af vindmøllevinger (Static Structural Testing of Rotorblades).

S.I. Andersen, presented at the meeting: Vindmøllevinger og materialevalg, Dansk Selskab for materialeprøvning og -forskning, Risø National Laboratory, 20 March 1986. To appear in Materialenyt.

The principles for the static structural testing of glass/polyester rotor-blades are summarized, and it is shown that a series of 2-3 load cases with only 1 load supplied at different positions is a valid approximation to the assumed distributed design load. The test results from 5 different rotor-blades are presented and compared with the present design criteria.

Strain Gauge Measurements on Anisotropic Materials.

S.I. Andersen, presented at a Seminar on Effects of Defects in Composites, DFVLR, Köln, 21-24 October 1986. (Not available).

The problems involved in the application of resistance foil strain gauges on anisotropic materials are surveyed with emphasis on the correction of errors introduced due to the sensitivity of the gauges.

Thermal Decomposition of (Cerium, Gadolinium)- and (Cerium, Europium)-Carbonates: Compositional and Structural Changes, and Kinetics.

J.J. Bentzen, P.L. Husum and O. Toft Sørensen, presented at the World Congress on High Tech Ceramics, Milano, 23-29 June 1986. Available on request (14 pp). (Proceedings to be published).

The existence of a complete solid solution was found for the system $(Ce_{1-x}RE_x)_2O(CO_3)_2 \cdot H_2O$, RE = Gd, Eu, $0 \leq x \leq 1$. The X-ray diffraction patterns of $M_2O(CO_3)_2 \cdot H_2O$, M = Ce, Gd, Eu, are given. The decomposition of $(Ce_{1-x}RE_x)_2O(CO_3)_2 \cdot H_2O$ to $Ce_{1-x}RE_xO_{2-x/2}$ took place at one to three different temperature levels depending on x and the heat-treatment. The lower decomposition temperature increased with x to a maximum for x = 0.9. For Ce-, Eu-, and Gd-carbonate the rate determining step at low temperature was evaluated as nucleation followed by two-dimensional growth, and those of the second and third temperature levels for the Eu- and Gd-carbonates were estimated as two-dimensional nuclei growth. The oxide crystallites formed were initially ~ 100 Å and grew into cubes and pyramid-cubes reaching sizes of 0.1-0.3 μm at 1000°C. Very high surface area (~ 100 m^2/g) oxide powders could be obtained by calcination at low temperatures.

Silver Saving by Substituting Brazing Alloys with 30-50% Ag for CuP Alloys with 0-15% Ag.

J. Christensen and P. Brøndsted, presented at an EEC conference, Bruxelles, 11. December 1986. Available on request. (Proceedings to be published).

The properties of the insufficiently documented copper joints brazed with the various AgCuP-brazing alloys have been investigated - especially creep and fatigue data have been obtained using standard procedures for testing brazed joints. Also the fatigue strength of the as cast brazing alloys have been measured since they could be present as a continuous phase in the wider gaps - 0.1 mm, and thus be determining for the properties of the brazed joints. The creep and fatigue strength measured showed that the strengths of the joints brazed with the various brazing alloys as well as the strength of the alloys themselves were all well above the strength of copper heat treated according to standard brazing procedures. Results of direct use for practice were demonstrated by the fatigue tests carried out on standard brazed joints of normed copper tubes. It was found that brazed joints with irregularities and overlap length of only 30% of the normed always fractured in the copper tube. With respect to the creep and fatigue properties measured the normally used 30-50% Ag-bearing brazing alloys can be substituted by the 0-15% AgCuP alloys. Thereby the silver consumption can be reduced and economical savings obtained by industry.

Standardization Problems in Neutron Radiography of Nuclear Reactor Fuel.

J.C. Domanus, presented at the 8th International Conference on NDE in Nuclear Industry, Kissimee, 17-20 November 1986. Available on request (8 pp). (Proceedings to be published).

Although ASTM has published three standards for standard methods and practices for neutron radiography, there is still a need for special standards in the field of neutron radiography of nuclear fuel. As in other fields of industrial radiography, the radiographic image quality of neutron radiographs should be controlled by the use of image quality indicators (IQI) and when classifying defects revealed by neutron radiography one ought to have the possibility of using standard reference radiographs. The Euratom Neutron Radiography Working Group (NRWG) tries to solve these standardization problems. In 1984 it published "Reference Neutron Radiographs of Nuclear Reactor Fuel". Because ASTM indicators are not suitable for neutron radiography of nuclear fuel, an attempt will be made to design a special IQI, based on the results reached with the calibration fuel pin.

Effect of Metallurgical Parameters on the Textural Development During Cold Deformation of Aluminium and Aluminium Alloys.

N. Hansen and D. Juul Jensen, presented to the American Institute of Mining and Engineering, The Metallurgical Society, Orlando, Florida, 6 October 1986. (To be published).

The effect of grain size and of large and small particles on the deformation textures of aluminium and other fcc metals was discussed.

EUREKA/EUROMAT.

N. Hansen, presented to Ingeniørsammenslutningen, København, 18 February 1986. (Not available).

The possibilities for Danish participation in the materials programme (EUROMAT) within EUREKA was discussed.

Nucleation and Grain Growth.

N. Hansen, presented at the Materials Department, Norges Tekniske Højskole, Trondheim, 26 May 1986. (Not available).

Nucleation and grain growth during and after recrystallization was discussed with special emphasis on the effect of large and small particles.

Recrystallization in Commercial Aluminium.

N. Hansen, presented to Årdal og Sunndal Verk a.s., Sunndalsøra, 27 May 1986. (Not available).

The recrystallization behaviour of commercially pure aluminium was discussed with special reference to nucleation, grain growth and texture.

Influence of Grain and Sub-Grain Boundaries on Void Formation and Growth in Aluminium Irradiated with Fast Neutrons.

A. Horsewell and B.N. Singh, presented at the 13th International Symposium on the Effects of Radiation om Materials, Seattle, 23-25 June, 1986. To appear in ASTM Special Technical Publications. Available on request (24 pp.).

High purity aluminium was irradiated at 120°C to fast neutron fluences between $2 \cdot 10^{21}$ and $5 \cdot 10^{24}$ n/m^2. Variations in void density and void size in the vicinity of grain and sub-grain boundaries have been analysed. The results document that vacancy accumulation is enhanced in a wide zone up to 15 μm from grain boundaries. The magnitude of the enhanced vacancy accumulation varies as a function of distance from the grain boundary. The presence of sub-grain boundaries enhances the vacancy accumulation within the subgrain and the vacancy accumulation varies markedly as a function of sub-grain size. The results are discussed in terms of the consequences of an initial vacancy concentration gradient near grain boundaries on further vacancy accumulation. The maintenance and evolution of vacancy supersaturation gradients is considered in terms of transport and screening of interstitials during vacancy accumulation in the peak zone.

Computer Controlled Forced Stepwise Isothermal Analysis.
P.L. Husum and O. Toft Sørensen, presented at the 10th Nordic Symposium on Thermal Analysis and Calorimetry, Bergen, Norway, 20-22 August 1986. (Not available).

A new version of the Stepwise Isothermal Analysis technique is described in which the temperature is forced to increase in predetermined steps during a reaction. Data obtained by this technique can be used to determine not only the controlling mechanism but also the activation energy and the pre-exponential factor in the Arrhenius equation. As a demonstration of the advantages of this technique the results obtained in a kinetic study of the thermal decomposition of cerium carbonate in different atmospheres is described.

Effect of Thermal Processing and Initial Microstructure on Recrystallization of Aluminium.
D. Juul Jensen and N. Hansen, presented at the ASM Materials Week, Orlando, Florida, 9 October 1986. (Not available).

The recrystallization process in pure and commercially pure aluminium has been studied by in-situ neutron diffraction texture measurements and various microscopical techniques. The sample parameters were the initial grain size and the degree of deformation. The recrystallization process was followed during isothermal annealing at different temperatures as well as during more complex annealing treatments. The effect of the sample parameters and the annealing procedure on the microstructural and textural development is discussed as well as the kinetics of the recrystallization process.

Statistical Analysis of the Dimensional Measurements Under the NRWG Test Program.

Henrik Jensen, presented to Euratom Neutron Radiography Working Group, Risø National Laboratory, Roskilde, 28 October 1986. (Not available).

Characterization of Vacancy and Vacancy-Gas Agglomerates in Aluminium Irradiated with Medium Energy Protons by Positron Annihilation.

K.O. Jensen, M. Eldrup, B.N. Singh, A. Horsewell, M. Victoria and W.F. Sommer, presented at the conference Vacancies and Interstitials in Metals and Alloys, Berlin, 14-19 September 1986. To appear in Materials Science Forum.

Positron annihilation and electron microscopy investigations of aluminium samples irradiated with 600 or 800 MeV protons or with neutrons have been carried out. The samples contain helium bubbles or voids of various sizes. This was used to determine the positron specific trapping rate into the cavities as a function of radius. Using this function and a theoretical estimate of the helium density in the bubbles based on the trapped-positron lifetime it is shown how the average bubble size and density can be derived from the positron lifetime data. Finally, the shapes of the angular correlation curves for positrons trapped in bubbles are derived and correlated with the He density in the bubbles.

Positron Annihilation in He Bubbles in Al.

K.O. Jensen, R.M. Nieminen, M. Eldrup and B.N. Singh, presented at the 6th General Conference of the Condensed Matter Division of the European Physical Society, Stockholm, 22-25 March 1986, and at Dansk Fysisk Selskab Faststofsektionen/ Atomfysiksektionens Forårsmøde '86, Nyborg, 20-21 May 1986. (Not available).

The state of a positron trapped at an Al-He interface has been studied theoretically. The distribution of He across the interface was calculated by the molecular dynamics technique. The results demonstrate a concentration enhancement over the average He concentration close to the Al surface at average He densities above $4.5 \cdot 10^{22}$ cm^{-3}. The influence of the He atoms on a positron trapped in the image potential well outside the Al surface was determined in the corrugated mirror model. The annihilation rate of an interface trapped positron is increased compared to the clean surface annihilation rate due to annihilation with He electrons.

The theoretical results are compared with experimental results for positrons trapped in He bubbles in annealed 600 MeV proton irradiated Al samples. Independent estimates of the He concentration in the bubbles are obtained from the bubble sizes (determined by TEM) assuming the bubbles to be in thermal equilibrium. The agreement between experiment and the theoretical relationship between He density and positron lifetime is very good, thus supporting the proposal that positron annihilation can be used to determine He densities inside He bubbles in metals.

Positrons at Internal Surfaces: Helium Bubbles in Aluminium.
K.O. Jensen, R.M. Nieminen, M. Eldrup and B.N. Singh, presented at an International Workshop on Slow Positron Beam Techniques, Norwich, 7-10 July 1986. Available on request (16 pp.).

Experimental and theoretical results are presented for positrons in He bubbles in Al with emphasis on those aspects which are related to the interaction between the positron and the bubble surface. For Al 600 MeV proton-irradiated at 600-700 K a narrow ACAR peak indicates a Ps-like positron state in the He bubbles possibly localized at the bubble surface. In samples irradiated at 400-500 K measured positron lifetimes can be related to He densities inside bubbles. Theoretical calculations for positrons trapped in the image-potential well at the bubble surface (i.e. at the He-Al interface) have been made and a relation is obtained between the positron lifetime and He densities in bubbles.

Measurement of Internal Stresses by Neutron Diffraction Using a Position Sensitive Detector.
T. Leffers, T. Lorentzen, D. Juul Jensen and J. Kjems, presented at the International Conference on Residual Stresses, Garmisch-Partenkirchen, 15-17 October 1986. Proceedings to be published.

It is well known that neutron diffraction can detect bulk internal stresses because of the high penetration power of neutrons in most materials - whereas only surface stresses can be detected with X-ray diffraction. In this paper it is pointed out that there are other significant advantages with neutron diffraction. The paper also deals with the difficulties connected with the effects of elastic anisotropy and texture common to both diffraction methods. Two new experimental concepts for the measurement of microscopic and macroscopic internal stresses with neutron diffraction are described.

Fiberforstærkede kompositmaterialer, egenskaber og dimensionering (Fibre-Reinforced Composite Materials, Properties and Dimensioning).

H. Lilholt and Aa. Lystrup, presented to Dansk Selskab for Bygningsstatik, Danmarks Tekniske Højskole, Lyngby, 18 February 1986. (Not available).

The basic properties of fibre composites and their constituents are reviewed, and different load conditions are discussed. Special emphasis is paid to long term properties (fatigue, creep), lamination theory, and design criteria. The principles in designing beams, columns, and plates are also discussed, and some applications and their fabrication methods are illustrated.

Metal matrix kompositmaterialer (Metal Matrix Composite Materials).

H. Lilholt and S. Feldager Hansen, presented at a FTU-Seminar, Danmarks Tekniske Højskole, 27-28 August 1986. Available on request (9 pp.).

The project on metal matrix composite materials is reviewed. The materials are aluminium with ceramic fibres (silicon carbide or aluminium oxide); the fabrication routes are pressure casting and powder metallurgy; the properties are thermal fatigue and creep.

Modelling of the Second and Third Stage Creep Rates of an Aligned Short Fiber Metal Matrix Composite.

M. Taya and H. Lilholt, presented at the ASME Wintermeeting, Anaheim, California, 10 December 1986. (Proceedings to be published).

A model is proposed for the creep rates of short fibre metal matrix composites the model is based on the exponential creep law for the matrix; the effect of fibre volume fraction and fibre length is studied. The effect of debonding at the fibre/matrix interface is considered and used to model the creep rate in the third stage.

Termoplast kompositmaterialer (Thermoplastic Composite Materials).

H. Lilholt and R. Talreja, presented at a FTU-Seminar, Danmarks Tekniske Højskole, 27-28 August 1986. Available on request (4 pp.).

The project on composite materials with a thermoplastic matrix is reviewed: The materials are polyether-ether-ketone (PEEK) with carbon fibres; the fabrication route is hot pressing; the properties are fatigue and creep.

Nye materialer og deres anvendelsesmuligheder. Plastbaserede kompositmaterialer (New Materials and Their Potential Applications. Composite Materials Based on Plastics).

Aa. Lystrup and H. Lilholt, presented to Produktionsteknisk Selskab, Risø, 27 February 1986. (Not available).

The basic properties of fibre composites and their consituents are reviewed and different load conditions are discussed. The principle in designing with fibre composites is also discussed, and special emphasis is paid to the correlation between the various fabrication methods and design possibilities.

Fission Gas Release Mechanisms Operating in Water Reactor Fuel in Power Transients.

M. Mogensen, P. Knudsen and C.T. Walker, presented at the IAEA International Symposium on Improvements in Water Reactor Fuel Technology and Utilization, Stockholm, 15-19 September 1986. Available on request (15 pp.). (Proceedings to be published).

Local Xe concentrations in grains and in grain boundary porosities were obtained as a function of relative radius on pellets of transient tested high burnup BWR and PWR fuel. The techniques used were X-ray fluorescence analysis (XRF) and electron probe micro analysis (EPMA). The PWR fuel had a burnup of 3.4% FIMA (31 GWd/tU) and was held for 28 h at a transient terminal level of 32 kW/m. The BWR fuel with 4.3% FIMA (39 GWd/tU) was held at the terminal level of 42 kW/m for 24 h. Considerable amounts of gas in the grain boundary porosities were found at certain radial positions, up to 40% of the Xe generated for the PWR and up to 20% for the BWR fuel. The Xe distribution within single grains in the PWR fuel was measured by EPMA.

On the basis of these observations, the mechanisms of transient fission gas release from high-burnup fuel are discussed. It is shown that for both fuel types the gas release is limited by the tunnel formation process and not by diffusion of gas from grain interior to the grain boundary.

Nucleation and Growth of LiCl Passivating Layers on Electrodes in Thionyl Chloride.

M. Mogensen, presented at the 15th International Power Sources Symposium, Brighton, UK, 8-11 September 1986. Available on request (11 pp.). Proceedings to be published.

The kinetics of LiCl layer formation on lithium and stainless steel electrodes were studied by means of ac-impedance. Li electrodes were followed from a few minutes to years of exposure in $SOCl_2$ solutions of 1.8 M $LiAlCl_4$ and 1.2 M $LiAlCl_4$ + 0.6 M SO_2AlCl_3. A stainless steel electrode was polarized 3.68 V to the free potential of Li using a potentiostat in order to get the current as a function of time. Also the ac-impedance of this electrode was measured. The results indicate that the LiCl film growth on Li in the early stages is controlled by a nucleation and growth type of mechanism whereas the LiCl production (electronic conductivity) is the rate determining step after about 2 days.

On the Rate Determining Step in Fission Gas Release at High Burnup.

M. Mogensen, P. Knudsen and C.T. Walker, presented at the Enlarged Halden Program Group Meeting, Sanderstølen, Norway, 2-7 March 1986. Available on request (16 pp.).

Radial Xe concentration profiles were measured by means of electron probe micro analysis (EPMA) and X-ray fluorescence (XRF) on two different types of transient tested high burnup fuel. Because the XRF measures all Xe retained (including the Xe in the grain boundary porosities) and EPMA detects the Xe-inside the grains only, it is possible to obtain the amount of Xe in the grain boundary porosities as the difference between the radial concentrations measured by the two techniques.

The samples investigated were PWR and BWR test fuels. Both fuels were transient tested at the end of life. The PWR fuel had a burnup of 3.4% FIMA (31 GWd/tU), it was transient tested to 32 kW/m and the hold time at the transient terminal level was 28 h. The BWR fuel had a burnup of 4.3% FIMA (39 GWd/tU) and had a transient terminal power of 42 kW/m for 24 h. Besides this the two fabrication routes were different.

Very different amounts of grain boundary gas were observed in the two cases. Both sets of observations indicate, however, that it is the formation and interlinkage of porosity at the grain boundary rather than the transportation of Xe from the grain interior which is the rate determining step. At a certain radial position in the PWR fuel for example, 35% of the Xe generated was retained inside the grains, 40% was retained in porosities and 25% was released to the internal void in the pin.

Properties of LiCl Layers on Li in Various $SOCl_2$ Solutions.

M. Mogensen, presented at the 3rd International Meeting on Lithium Batteries, Kyoto, 27-30 May 1986. To appear in Journal of Power Sources. Available on request (13 pp.).

The growth rate of LiCl solid electrolyte layers on Li was studied in neutral and acid $LiAlCl_4$-$SOCl_2$ solutions during periods of years. For SO_2-containing acid solution a small anodic load from coupling Li to stainless steel proved to be more important than the acidity. The ionic and electronic conductivities of the layers were derived from impedance measurements and passivation rates, respectively. In neutral solutions the Li^+ conductivity is decreasing with increasing layer thickness whereas this effect was not seen in the acid SO_2-containing solution. The factors influencing the growth rate of the LiCl layer are briefly analysed. It is concluded that increasing the ratio of ionic to electronic conductivity would be the most efficient way of improving the performance of the solid electrolyte.

Sputtering.

L. Nordentoft, presented at Dansk Forening for Materialografis Novemberseminar, Kolding, 20-21 November 1986. (Not available).

The principles of the sputtering technique and the advantages of thin films produced by rf-sputtering were reviewed.

Lavtrykslagring af naturgas ved adsorption (Low Pressure Adsorption Storage of Natural Gas).

A. Schrøder Pedersen, presented at Nordisk seminar om energi- og miljøforbedrende foranstaltninger i bybusser, Esbjerg Højskole, 9-10 December 1986. Proceedings to be published. Available on request (3 pp.).

The technical aspects of utilising adsorption of natural gas on surfaces of solids were reviewed.

The Formation of Hydride in Pure Magnesium Foils.

A. Schrøder Pedersen, J. Kjøller, B. Larsen and B. Vigeholm, presented at the International Symposium on the Properties and Applications of Metal Hydrides V, Maubuisson, France, 25-30 May 1986. To appear in J. Less-Common Met. Available on request (14 pp.).

In this work pure magnesium was rolled to foil thicknesses down to 20 μm. The foils were exposed to hydrogen in the pressure range 1-3 MPa and temperatures between 650 and 700 K. The hydride formation was followed by gravimetry and by microscopy. The effect of foil thickness on the hydride formation is described and the mechanism and kinetics of hydride formation are discussed. It is concluded that the chemical reaction between magnesium and hydrogen is the rate limiting step for the growth of the nuclei and that the transport processes are fast.

Composite Modelling of Tensile and Cyclic Plasticity in Copper.
O. Bøcker Pedersen, presented at Institut for Fysikalsk Metallurgy, Norges tekniske Højskole, Trondheim, 21 March 1986. (Not available).

The reverse microflow associated with the Bauschinger effect in copper strained into stage II is characterized experimentally and analyzed in terms of the theory of obstacle-controlled flow and established composite theory. It is concluded that the flow mechanisms in the hard and soft regions cannot be specified separately, the flow resistance in the soft regions arises from the constraint on long-range glide imposed by the forest concentrated in the hard regions. The problem of the relation between plastic strain and dislocation microstructure remains unsolved.

Characterisation and Uses of Solid Electrolytes.
F.W. Poulsen, presented at the Department of Chemistry and Applied Chemistry, University of Salford, UK, 19 November 1986. (Not available).

A multi-instrumental approach is taken at Risø in order to characterise solid electrolytes. The targets for this research are: oxygen sensors and HT-fuel cells based on oxygen ion conductors, and lithium batteries with solid lithium ion conductors. Impedance spectroscopy, SEM and neutron diffraction are the more important methods. Attempts are made to study the materials both as single crystals, polycrystals and thin films. New types of solid electrolytes and model systems are also described.

Effect of Different Internal Surfaces in Composite Li-Electrolytes.
F.W. Poulsen, presented at the 3rd International Meeting on Lithium Batteries, Kyoto, 27-30 May 1986. To appear in Journal of Power Sources.

A linear increase of the conductivity of LiI-alumina composite electrolytes with increasing specific surface area (2.4-260 m^2/g) of the alumina is demonstrated. Part of the enhanced conductivity is probably due to normal doping of the LiI by the alumina. Replacing 25% of the LiI by LiBr did not change the conductivity. Replacing part of the LiI by Li_3N had a detrimental effect.

Diffusion Mechanisms for Enhanced Vacancy Accumulation Near Planar Sinks.

A.J.E. Foreman, B.N. Singh and A. Horsewell, presented at the International Conference on Vacancies and Interstitials in Metals and Alloys, W. Berlin, 14-19 September 1986. To appear in Materials Science Forum. Available on request (6 pp.).

Both the nucleation and growth of cavities are appreciably enhanced in a relatively wide (of the order of 20 cavity spacings) band near planar sinks, e.g. grain boundaries. The vacancy accumulation peaks at a distance of about 10 cavity spacings from the boundaries. We speculate here on the nature of the cavity enhancement arising from various modes of interstitial transport. We consider the conventional three-dimensional and the anisotropic diffusion of self-interstitials, and dynamic transport of atoms into boundaries by replacement sequences or long range channelling. Predictions of these calculations are compared with experimental results.

Microstructural Changes in Commercial Aluminium Alloys due to Irradiation with 800 MeV Protons.

B.N. Singh, W.F. Sommer, W. Lohmann and A. Ribbens, presented at the 13th International Symposium on the Effects of Radiation in Materials, Seattle, 23-25 June, 1986. To appear in ASTM Special Technical Publications. Available on request (23 pp.).

Commercial Al-Mg and Al-Mg-Si alloys were irradiated in the Los Alamos Meson Physics Facility (LAMPF) with 800 MeV protons. The irradiation experiment was carried out at a temperature between 40 and 100°C to a dose level of $\leq$ 0.2 dpa. Post-irradiation microstructure and mechanical properties of these alloys have been investigated. For comparison, a parallel investigation was conducted on unirradiated reference specimens as well as on unirradiated annealed (981 h at 100°C, corresponding to time at temperature during irradiation) specimens. It was found that the cold-work microstructure in Al-Mg alloy survives the thermal annealing treatment but dissolves during irradiation already at a dose level of $\leq$ 0.1 dpa. In the Al-Mg-Si alloy Mg_2Si type precipitates also survive the thermal annealing but dissolve during irradiation at a low dose

level of $\lesssim$ 0.1 dpa. Post-irradiation mechanical testing demonstrated that the strength of these alloys is drastically reduced due to irradiation. The observed irradiation softening can be understood in terms of the observed microstructural changes caused by irradiation.

Nucleation, Growth and Distribution of Cavities in the Vicinity of Grain Boundaries in Aluminium Irradiated with 600 MeV Protons.
M. Victoria, W.V. Green, B.N. Singh and T. Leffers, presented at the 13th International Symposium on the Effects of Radiation on Materials, Seattle, 23-25 June, 1986. To appear in ASTM Special Technical Publications. Available on request (24 pp.).

High-purity aluminium (99.9999%) was irradiated with 600 MeV protons with a damage rate of 3.5 x 10^{-6} dpa/s and a helium generation rate of 214 appm/dpa. The irradiation experiments were carried out at temperatures in the range 120 to 140°C to doses up to 5 dpa. Transmission electron microscopy on irradiated specimens showed that voids are formed only in a narrow band in the vicinity of the void denuded zone along grain boundaries. Both the density and the size of voids in this band vary as a function of distance from the denuded zone. Changes in irradiation dose and temperatures do not seem to affect the spatial distribution of voids in the band. These results are compared with the results obtained on high-purity aluminium irradiated with fast neutrons. The bubble formation behaviour in the grain interior and in the band containing voids is briefly described. The effect of bubble formation on the growth behaviour of voids is discussed.

On Transport of Helium to Grain Boundaries During Irradiation.
B.N. Singh and A.J.E. Foreman, presented at the 13th International Symposium on the Effects of Radiation on Materials, Seattle, 23-25 June 1986. Also published as Risø-M-2612.

Iltmåling baseret på avanceret keramik (Measurement of Oxygen Based on Advanced Ceramics).
O. Toft Sørensen, presented to Foreningen for Saltsmelteforskning, Danmarks Tekniske Højskole, Lyngby, 6 June 1986. (Not available).

The lecture covered: principle of electrochemical oxygen sensors, materials (electrolytes and electrodes) and applications of oxygen sensors.

Keramik for oxygen-sensorer og brændselsceller (Ceramics for Oxygen Sensors and Fuel Cells).

O. Toft Sørensen, presented at Symposium om Avanceret Teknisk Keramik, Danmarks Tekniske Højskole, Lyngby, 22 May 1986. (Not available).

General properties and applications of oxygen ion conductors in oxygen sensors and fuel cells were presented and discussed.

Oxidbaserede avancerede keramiske materialer (Oxide-Based Advanced Ceramic Materials).

O. Toft Sørensen, presented to Procesteknisk Selskab, Ingeniørsammenslutningen, København, 20 March 1986. (Not available).

General properties and examples of applications were presented and discussed for two types of advanced ceramic materials: zirconia toughened ceramics, which today can be manufactured with high strength and fracture toughness, and oxygen conducting oxides.

Research and Development in Advanced Ceramic Materials in Denmark.

O. Toft Sørensen, presented at Teknologisk Institut, Reykjavik, 29 May 1986. (Not available).

After a short introduction about Risø National Laboratories, the present R&D work in Denmark in advanced ceramic materials were discussed. Particularly two types of material were covered: zirconia toughened ceramics (structural ceramics) and oxygen ion conducting oxides. Finally a survey was presented about the EEC programmes supporting such R&D work in Europe.

Some Recent Developments at Risø National Laboratory in Studies of Nonstoichiometric Oxides and in the Field of Thermal Analysis.

O. Toft Sørensen, presented at Tokyo Institute of Technology, Yokohama, 12 September 1986, and National Defence Academy, Yokosuka, Japan, 18 September 1986. (Not available).

After a short introduction about Denmark and Risø a survey was presented of the author's work in two fields:

1. nonstoichiometric oxide systems - CeO_{2-x},$Fe_{1-y}O$.
2. thermal analysis - stepwise isothermal analysis techniques.

Some Recent Developments in Thermal Analysis in Denmark.
O. Toft Sørensen, presented at the 10th Nordic Symposium on Thermal Analysis, University of Bergen, Norway, 20-23 August 1986. (Not available).

Danish industries and research institutions using thermal analysis were presented. As an example of work involving development of new techniques, the new stepwise isothermal analysis technique developed at Risø was discussed.

Pilot Plant Production at Risø of LEU Silicide Fuel for the Danish Reactor DR 3.
P. Toft, J. Borring and E. Adolph, presented at the 1986 International Meeting on Reduced Enrichment for Research and Test Reactors, Argonne National Laboratory, 3-6 November 1986. (Not available).

A pilot plant for fabricating LEU silicide fuel elements has been established at Risø National Laboratory. Three test elements for the Danish reactor DR3 have been fabricated, based on 19.75% enriched U_3Si_2 powder that has been purchased elsewhere. The pilot plant has been set up and test elements fabricated without any major difficulties.

Elements of Hydride Formation Mechanisms in Near Spherical Magnesium Powder Particles.
B. Vigeholm, K. Jensen, B. Larsen and A. Schrøder Pedersen, presented at the International Symposium on the Properties and Applications of Metal Hydrides V, Maubuisson, France, 25-30 May 1986. To appear in J. Less-Common Met. Available on request (14 pp.).

Experiments on a large number of magnesium powders varying in particle size, morphology, purity and surface oxidation have shown that most of these parameters influence the kinetics of the hydride formation. Although no single step in the reaction will in general be rate determining we have in a previous investigation been able to assign a nucleation and growth model to the initial

hydriding of an atomized powder. This powder of nearly spherical particles (d~90 μm) with a thin oxide coating has been used in this further investigation of the nucleation and growth mechanisms. It is found that the nucleation is rate determining in the initial hydriding only and that the growth takes place entirely by interface migration of hydrogen from the particle surface. The pressure/nucleation relation and impurity effects on the ultimate degree of reaction are discussed.

Energy Storage Applications of Magnesium.

B Vigeholm, presented the Conference on Magnesium Technology, The Institute of Metals, The Royal Society, London, 3-4 November 1986. Proceedings to be published. Available on request (15 pp.).

This paper outlines the concept of energy storage in metal hydride with emphasis on the magnesium/magnesium hydride system. An attempt is made to survey the magnesium and magnesium alloy hydride application as it may be inferred from operating, planned and intended programmes mainly involving other hydrides. The overview encloses hydrogen storage, heat storage, heat pumps, hydrogen recovery and refining.

Lagring af brint som metalhydrider (The Storage of Hydrogen in Metal Hydrides).

B. Vigeholm, presented at the meeting Brint: fremstilling, lagring og anvendelse (Hydrogen, Production, Storage and Applications). Askov Højskole, Vejen, 29 November 1986. Available on request (20 pp.).

Storage of hydrogen in metal hydrides is seen in the light of future demands for non-polluting energy application. The state of the art and expected implementations are outlined.

STAFF OF THE DEPARTMENT
(as at 31 December 1986)

Niels Hansen, Head of Department

Scientific Staff

Adolph, E.
Andersen, Sv.I.
Bagger, C.
Bentzen, Janet
Bilde-Sørensen, J.B.
Borring, J.
Brøndsted, P.
Carlsen, Hans
Christensen, Jørgen
Debel, C.P.
Domanus, J.
Eldrup, M.
Gundtoft, H.E.
Horsewell, A.
Husum, P.L. 1)
Jensen, Dorthe Juul
Johansen, Bjørn S.
Knudsen, Per
Leffers, T.
Lilholt, H.
Lisiecki, L.L. 2)
Lorentzen, T. 3)
Lystrup, Aa.
Mogensen, Mogens
Nordentoft, L.
Ottosen, N. Saabye 4)
Pedersen, A. Schrøder
Pedersen, O. Bøcker
Poulsen, Finn W.
Rørbo, K.
Singh, B.N.
Sørensen, O. Toft
Toft, P.
Toftegaard, H.L.
Vigeholm, B.

Technical Staff

Adrian, F.
Andersen, Axel B.
Aukdal, J.A.
Berthelsen, Brian
Bülow-Christensen, C.J.
Christensen, Sv.E.
Cooper, D.
Dreves, P.
Frederiksen, H.
Friedrichsen, U.J.
Gravesen, Niels N.
Hersbøll, B.
Jacobsen, Mogens L.
Jakobsen, Mogens M.
Jensen, Finn
Jensen, Knud
Jensen, Palle V.
Jensen, Søren D.
Jespersen, John
Kjep, T.
Kjøller, J.
Klitholm, C.
Koustrup, Jan
Larsen, Bent
Larsen, Jan
Larsen, Kjeld J.C.
Lindbo, J.
Mikkelsen, Claus
Nielsen, Ove
Nielsen, Palle
Nielsen, Torben
Nilsson, H.
Olesen, Preben B.
Olsen, Benny P.
Olsen, Bent
Olsen, Henning
Olsen, Ole
Olsson, Jens
Paulsen, Henrik
Pedersen, Børge
Pedersen, Knud E.
Pedersen, Niels J.
Petersen, Jan H.
Rasmussen, Allan
Rasmussen, Henrik
Robl, Steen
Sandsted, K.
Strauss, T.R.
Sørensen, Erling
Thomsen, Leif
Aagesen, Sv.

Office Staff

Arildsen, Nina
Dreves, Elsa
Frederiksen, Anelise
Jørgensen, Lone
Lauritsen, Grethe W.
Szöts, Anni
Sørensen, Eva

1) Post-graduate student from the Technical University of Denmark
2) Visiting Scientist from Northwestern University, Evanston, USA
3) Post-graduate student from Aalborg University Centre, Denmark
4) Leave of absence

Risø National Laboratory **Risø - R - 547**

Title and author(s)

Metallurgy Department Progress Report
for the Period 1 January to 31 December 1986

Edited by A. Schrøder Pedersen and
J.B. Bilde-Sørensen

Date April 1987

Department or group Metallurgy Department

Groups own registration number(s)

Project/contract no.

Pages 96 Tables Illustrations 16 References

ISBN 87-550-1311-2

Abstract (Max. 2000 char.)

The activities of the Metallurgy Department at Risø during 1986 are described. The work is presented in four chapters: General Materials Research, Technology and Materials Development, Chemical and Electrochemical Energy Research and Development, and Fuel Elements. A survey is given of the Department's participation in international collaboration and of its activities within education and training. A list (with abstracts) of publications and lectures by the staff during 1986 is included.

Descriptors - INIS;
BIBLIOGRAPHIES; ELECTROCHEMISTRY; FUEL ELEMENTS; MATERIALS TESTING; METALLURGY; NONDESTRUCTIVE TESTING; RESEARCH PROGRAMS; RISØ NATIONAL LABORATORY

Available on request from Risø Library, Risø National Laboratory, (Risø Bibliotek, Forskningscenter Risø),
P.O. Box 49, DK-4000 Roskilde, Denmark.
Telephone 02 37 12 12, ext. 2262. Telex: 43116, Telefax: 02 36 06 09

Sales distributors:
G.E.C. Gad Strøget
Vimmelskaftet 32
DK-1161 Copenhagen K, Denmark

Available on exchange from:
Risø Library, Risø National Laboratory,
P.O.Box 49, DK-4000 Roskilde, Denmark

ISBN 87-550-1311-2
ISSN 0106-2840